Quality Assurance Project Plan

ORIGINAL

Remedial Investigation/ Feasibility Study

Crab Orchard National Wildlife Refuge

U.S. Fish and Wildlife Service
U.S. Department of Interior
Marion, Illinois
and
Sangano-Weston, Inc.
Atlanta, Georgia

November 1986

O'BRIEN & GERE

Addendum No. 3

QUALITY ASSURANCE PROJECT PLAN
CRAB ORCHARD NATIONAL WILDLIFE PROJECT PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

ATTACHMENT 3

Procedures for Fish

Preparation and Analysis

Procedure for Pesticide/PCB
Determination in Fish

1.0 ANALYTICAL PROTOCOL REQUIRED

1.1 Grinding of Tissue and % Lipids - See Attachment 3-1.

1.2 Extraction for BNA and Pest/PCB - see Attachment 3-2, Method 3540 "Soxhlet Extraction". SW846 EPA Test Methods for Evaluating Solid Wastes, 2nd edition.

1.3 U.S. EPA-CLP "Statement of Work for Organics Analysis, Multi-Media/Multi-Concentration", 7/85 Revision.

2.0 SPECIAL TECHNICAL INSTRUCTIONS

2.1 Protect All Samples and Extracts from Light.

2.2. Sample Treatment

2.2.1 Sample will be shipped frozen. For preservation and ease in handling, keep frozen until analyzed. Once tissue specimen is processed into ground tissue, keep frozen until analyzed.

2.2.2 Optimally, a minimum of 5 specimens will be composited into one sample. Do not mix species, ie. composite 5 carp, but not 1 sunfish, 2 black bullhead and 2 carp in the same composite.

2.2.3 A minimum of 10 g ground tissue must be reserved for inorganic analysis.

2.3 Storage

2.3.1 The contractor is required to store (frozen), up to one year after data submission, all unanalyzed portions of samples submitted for analysis. Extracts will be stored in bottles/vials with teflon-lined septa and maintained at 4°C. Digestates will be stored in acid-washed bottles.

2.3.2 The Contractor must obtain approval from the Project Officer for authorization to dispose of samples/extracts/digestates or provide same to U.S. EPA within 7 days after a request by the Project Officer or the Sample Management Office (SMO).

2.4 Control Spikes

2.4.1 In addition to field specimens, the laboratory will receive a "clean" tissue sample to be used for preparation of control spikes. This "clean" sample will

consist of non-contaminated fish specimens obtained from a controlled environment, which the laboratory will composite/grind/homogenize in the same manner described for other tissue specimens (see Attachment 3-1), and store frozen. This sample will be referred to as the control matrix, and will be applicable to all animal analyses: small mammals, earthworms, crayfish, fish, and snapping turtles.

2.5 Sample Analysis

2.5.1 The ground tissue will be prepared/analyzed per CLP Organics SOW requirements for low-level soil/sediment samples.

2.5.2 The laboratory will determine and report % liquids (see Attachment 3-1). Sample results will be reported on wet weight bases.

2.5.3 Five-gram sample aliquots will be used.

2.5.4 Surrogate spikes and matrix spikes for Pest/PCB's will be added to the 5.0g sample per CLP requirement for 30g soil samples, so that extract concentrations will be the same as CLP, even though the sample concentration (ug/g) will differ from CLP.

For spiking Pest/PCB's blend/homogenize 5.0 g of ground tissue with a Tissuemizer. Blend ground sample with an equal weight of anhydrous sodium sulfate. Break up the caked material with a spatula and place in either a glass or paper extraction thimble. Spike with required matrix spike and/or surrogate spike compounds. Store the thimble containing the spiked sample 8 hours at ambient temperature to allow equilibration (can be stored on the Soxhlet or in an appropriately cleaned jar with a Teflon-lined cap). Proceed with extraction (see Attachment 3-2).

2.5.5 Extraction for Pest/PCB's will be as specified in Method 3540, Soxhlet extraction (see Attachment 3-2). The extract is cleaned-up/analyzed per CLP according to procedures in the Organics SOW.

2.5.6 GPC clean-up is required (refer to section in SOW). Note: samples will have high liquid content compared to soils. Use % liquids determination to prevent over-loading the GPC system. GPC model manufactured by ABC Labs (Columbia, MO) system will handle liquid concentrations up to 0.2 g/ml. Check with manufacturer for maximum liquid content if other vendors are used.

Use the full 5.0 ml aliquot sample extract for GPC clean-up. If liquid content is too high, GPC partial

volumes to equal 5.0 ml and combined the clean extracts.

3.0 ANALYTICAL RESULTS REQUIRED

3.1 Full CLP data/documentation deliverable, paginated; refer to deliverables section of the CLP.

3.2 Results of daily control spikes run with field specimens, reported on Form I and MS/MSD Form III annotated to read control spike/control spike duplicate.

4. OTHER

4.1 Quality Control Requirements will be per the CLP Organics CLP. In addition to the specified method blanks, matrix spikes and matrix spike duplicates, the control matrix will be spiked in the same manner (ie. same mix, concentration levels) as the method blank, MS and MSD to produce "control blank", "control spike", and "control spike duplicate" for daily analyses.

An MS/MSD will be done on each type of tissue. For example, for 20 samples consisting of moles, fish, worms, and batch will contain:

1) method blank
2) control blank
3) control spike/control spike duplicate
4) matrix spike and matrix spike duplicate for moles
5) matrix spike and matrix spike duplicate for fish
6) matrix spike and matrix spike duplicate for worms
7) up to 20 samples (field specimens other than QC samples)

Pesticides will contain all single component HSL compounds. PCB spikes will contain PCB 1242. Control spikes for daily analyses will be per CLP for matrix spikes.

ATTACHMENT 3-1

SAMPLE PREPARATION

Grinding

The Hobart Stainless steel meat grinder (or equivalent) is cleaned and rinsed with methanol and hexane. Dry ice is ground up and forwarded to the Inorganic section as an Inorganic Method Blank. Dry ice alone is ground and labelled as an Organic Method Blank. The fish is placed on a chopping block and chopped with either a cleaver or butcher saw into 2-3 inch cubes. The medium grinder sieve plate is inserted into the meat grinder and the fish cubes are ground. The smallest sieve is then inserted and the ground fish re-ground and collected in a stainless steel pan. The ground fish is mixed thoroughly with a spatula. At least a pint is placed and stored in a tared glass jar with a foil lined cover. This portion is to be extracted for organic compounds. Another portion (10 g) is placed in an acid washed glass jar for metals and analysis, if needed. Both portions should be kept frozen until analyzed.

Lipid Determination

The amount of extractable lipid is determined because the GPC system becomes inefficient when separating contaminants from lipids if the amount of lipid present in the extract is greater than 0.20 g/ml. The percent lipid can be a relative indicator of the overall condition of the fish.

Pipette a volume of sample extract sufficient to represent the equivalent of one gram of tissue into a preweighed and numbered aluminum drying pan (e.g., 10 g tissue extract in 10 ml solvent equivalent of 1 g/m). Place the pans in a fume hood (fan off and door closed) for 20 hrs to remove the solvent by evaporation. The percent extractable lipid is computed from the weight of lipid remaining in the pan.

EXTRACTION PROCEDURES

Scope and Application

Method 3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent. Subsequent cleanup and detection are described in the organic analytical method that will be used to analyze the extract.

Summary of Method

The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. Methylene chloride should be employed when a solvent is not specified. The extract is then dried and concentrated, and either cleaned up further or analyzed directly by the appropriate measurement technique.

Interferences

A procedural blank should be performed for the compounds of interest prior to the use of this method. The level of interference must be below the method detection limit before this method is used on actual samples.

More extensive procedures than those outlined in this method may be necessary for reagent purification.

Procedures for the removal of interfering compounds coextracted with target compounds are described in the organic analytical method that will be used to analyze the extract.

Apparatus and Materials

1) Soxhlet extractor: 40-mm I.D., with 500-ml round-bottom flask.

2) Kuderna-Danish appartus with three-ball Snyder column.

3) Chromatogrpahic column: Pyrex, 20-mm I.D., approximately 400 mm long, with coarse-fritted plate on bottom and an appropriate packing medium.

4) Glass or paper thimble or glass wool to retain sample in Soxhlet extraction device. Should drain freely and may require purification before use.

5) Boiling chips: Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

6) Rheostate controlled heating mantle.

Contract Required Detection Limits (CRDL)
for Fish

Pesticides	Detection Limits ug/g
101. alpha-BHC	0.02
102. beta-BHC	0.02
103. delta-BHC	0.02
104. gamma-BHC (Lindane)	0.02
105. Heptachlor	0.02
106. Aldrin	0.02
107. Heptachlor Epoxide	0.02
108. Endosulfan I	0.02
109. Dieldrin	0.04
110. 4,4'-DDE	0.04
111. Endrin	0.04
112. Endosulfan II	0.04
113. 4,4'-DDD	0.04
114. Endosulfan Sulfate	0.04
115. 4,4'-DDT	0.04
116. Endrin Ketone	0.04
117. Methoxychlor	0.20
118. Chlordane	0.20
119. Toxaphene	0.40
120. AROCLOR-1016	0.20
121. AROCLOR-1221	0.20
122. AROCLOR-1232	0.20
123. AROCLOR-1242	0.20
124. AROCLOR-1248	0.20
125. AROCLOR-1256	0.40
126. AROCLOR-1260	0.40

2 AC

Hexachlorobenzene
mirex

REFERENCES

1. Bishop, J.N., "Mercury in Fish", Ontario Water Resources Comm., Toronto, Ontario, Canada, 1971.

2. Boyle, H.W., et al., Adv. Chem. Serv., 60, 207 (1966).

3. Federal Register, Volume 41, No 232, p. 52780, Wednesday, December 1976.

4. Federal Register, Volume 44, No. 233, p. 69464, Monday, December 3, 1979.

5. Jones, J.W.; R. J. Gajan, K. W. Boyer; J. A. Fiorino; "Dry Ash - Voltammetric Determination of Cadmium, Copper, Lead, and Zinc in Foods." JOAC, 60, 825 (1977).

6. Jones, J.W.; R.J. Gajan; K. W. Boyer; J. A. Fiorino; "Dry Ash - Voltammetric Determination of Cadmium, Copper, Lead, and Zinc in Foods." JOAC, 60, 826. (1977).

7. Stalling, D.L.; R.C. Tindle; J.L. Johnson; "Cleanup of Pesticide and Polychlorinated Biphenyl Residues in Fish Extracts by Gel Permeation Chromatography." JOAC, 55, 32-38 (1972).

8. "Standard Methods for the Examination of Water and Wastewater", 14th edition (1975).

9. "EPA-CLP Inorganic Analysis: Multi-Media/Multi-Concentration", SOW No. 785, July 1985. Attachment 6: Method 245.5, Mercury in Sediments, pages D-52 through D-56. Annotated/Modified Method.

10. Handbook for Analytical Quality Control in Water and Wastewater Laboratories. U.S. Environmental Protection Agency, Technology Transfer, 1979.

11. "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", Physical and Chemical Methods Branch, EMSL-CI, revised October 1980. pages 27-29, 54. Primary Method: EPA 600/4-81-055. "EPA-CLP Inorganic Analysis: Multi/Media/Multi-Concentration", SOW No. 785, July 1985. Attachment #8: Method 335.2 (Sed.) CLP-M, Cyanide in Sediments, pages D-69 through D-80. Annotated/Modified Method.

12. "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268, 1978.

13. "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Technology Transfer. (1979).

Procedure for Metal
Determination in Fish

1.0 ANALYTICAL PROTOCOLS REQUIRED

1.1 Grinding of Tissue and % Lipids - See Attachment 3-1.

1.2 EPA 600/4-81-055 "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", Physical and Chemical Method Branch, U.S. EPA EMSL-CI, revised October 1980.

1.3 U.S. EPA-CLP "Statement of Work for Inorganics Analysis: Multi-Media/Multi-Concentration," SOW No. 785, July 1985.

1.4 Metals Digestion (perchloric acid digestion)-See Attachment 3-4.

2.0 SPECIAL TECHNICAL INSTRUCTIONS

2.1 Due to potential matrix effects, Zeeman AA Furnace is recommended for arsenic and selenium analyses.

2.2 Sample Treatment

2.2.1 Samples will be shipped frozen. For preservation and ease in handling, keep frozen until analyzed. Once tissue specimen is processed into ground tissue, keep frozen until analyzed.

2.2.2 Optimally, a minimum of 5 specimens will be composited into one sample. Do not mix species. ie. composite 5 carp, but not 1 sunfish, 2 black bullhead and 2 carp in the same composite.

2.2.3 A minimum of 10 g ground tissue must be reserved for inorganic analysis.

2.3 Storage

2.3.1 The Contractor is required to store (frozen), up to one year after data submission, all unanalyzed portions of samples submitted for analysis. Extracts will be stored in bottles/vials with Teflon-lined septa and maintained at 4°C. Digestates will be stored in acid-washed bottled.

2.3.2 The Contractor must obtain approval from the Project Officer for authorization to dispose of samples/extracts/digestates or provide same to U.S. EPA within 7 days after a request by the Project Officer or the Sample Management Office (SMO).

2.4 Control Spikes

2.4.1 In addition to field specimens, the laboratory will supply a "clean" tissue sample to be used for preparation of control spikes. This "clean" sample will consist of non-contaminated fish specimens obtained from a controlled environment, which the laboratory will composite/grind/homogenize in the same manner described for other animal specimens (see Attachment 3-1), and store frozen. This sample will be referred to as the control matrix, and will be applicable to all animal analyses: small mammals, earthworms, crayfish, fish, and snapping turtles.

2.5 Sample Analysis

2.5.1 The ground tissue will be prepared using the referenced digestion/distillation methods, followed by analysis using techniques specified in the U.S. EPA CLP Inorganic Analysis SOW 785.

2.5.2 The laboratory will determine and report % lipids (see Attachment 3-1). Sample results will be reported on wet weight bases.

2.5.3 Shellfish and bottom scavengers such as carp are expected to have higher levels of metals in their tissue than other fish.

2.5.4 Mercury will be digested/analyzed as described in Attachment 3-3.

2.5.5 All HSL metals other than mercury will be digested as described in Attachment 3-4, Perchloric Acid Digestion.

3.0 ANALYTICAL RESULTS REQUIRED

3.1 Full CLP data/documentation deliverables, paginated; refer to deliverables section of the SOW.

3.2 Results of daily control spikes and duplicates run with field specimens (Forms V and VI, respectively). Annotate Form V to specify control spike or matrix spike, as applicable. The matrix is "tissue". For spikes and duplicates also include the species, eg., tissue-fish, tissue-moles.

4.0 OTHER

I. Quality Control Requirements will be per the CLP Inorganic SOW 785. In addition to the specified method blank, matrix spike, and matrix spike duplicate, the control matrix used for method validation will be spiked in the same manner as the method blank and matrix spike. This will produce a "control blank"

and a "control spike" for daily analyses. The lab control sample referenced in the SOW will be the Metals in Fish from EMSL-CI.

A matrix spike and a matrix spike duplicate will be done on each type of animal. For example, for 20 samples consisting of moles, fish, and worms, the preparation batch will contain:

1) method blank
2) control blank
3) control spike
4) matrix spike and matrix spike duplicate for moles
5) matrix spike and matrix spike duplicate for fish
6) matrix spike and matrix spike duplicate for worms
7) lab control samples
8) up to 20 samples (field specimens other than QC samples).

Calibration curves will be a minimum of 3 concentration levels plus a blank for all analyses, including ICP. Calculate a linear regression, report correlation coefficient (R), slope, and y-intercept for each analyte. All reported results must be calculated from values obtained within the daily calibration range. R must be 0.996.

CRDL for Metals in
Fish Tissue Method

Element	Detection Limits ug/g
Cadmium	0.5
Lead	0.5
Mercury	0.2

ATTACHMENT 3-1

SAMPLE PREPARATION

Grinding

The Hobart Stainless steel meat grinder (or equivalent) is cleaned and rinsed with methanol and hexane. Dry ice is ground up and forwarded to the Inorganic section as an Inorganic Method Blank. Dry ice alone is ground and labelled as an Organic Method Blank. The fish is placed on a chopping block and chopped with either a cleaver or butcher saw into 2-3 inch cubes. The medium grinder sieve plate is inserted into the meat grinder and the fish cubes are ground. The smallest sieve is then inserted and the ground fish re-ground and collected in a stainless steel pan. The ground fish is mixed thoroughly with a spatula. At least a pint is placed and stored in a tared glass jar with a foil lined cover. This portion is to be extracted for organic compounds. Another portion (10 g) is placed in an acid washed glass jar for metals and analysis, if needed. Both portions should be kept frozen until analyzed.

Lipid Determination

The amount of extractable lipid is determined because the GPC system becomes inefficient when separating contaminants from lipids if the amount of lipid present in the extract is greater than 0.20 g/ml. The percent lipid can be a relative indicator of the overall condition of the fish.

Pipette a volume of sample extract sufficient to represent the equivalent of one gram of tissue into a preweighed and numbered aluminum drying pan (e.g., 10 g tissue extract in 10 ml solvent equivalent of 1 g/m). Place the pans in a fume hood (fan off and door closed) for 20 hrs to remove the solvent by evaporation. The percent extractable lipid is computed from the weight of lipid remaining in the pan.

ATTACHMENT 3-3

ANALYSIS OF FISH FOR MERCURY

Scope and Application

This method is used for determination of total mercury (organic and inorganic) in fish. Digest a weighed portion of the sample with sulfuric and nitric acid at 58°C. Follow by overnight oxidation with potassium permanganate at room temperature. Mercury is subsequently measured by the conventional cold vapor technique.

The range of the method is 0.2 to 5 ug/g but may be extended above or below the normal instrument and recorder control.

Sample Preparation

The sample may be prepared as described under "Sample Handling" or the special metal procedure may be used. A 0.2 to 0.3 g portion should be taken for each analysis. The sample should not be allowed to thaw before weighing.

Preparation of Calibration Curve

The tissue sample to be analyzed and the spiked control matrix fish tissue should be identically prepared. Prepare a calibration curve from values for portions of spiked control matrix fish tissue. For preparation of the calibration standards, choose a 5 g portion of fish and blend in a Waring blender.

Transfer accurately weighed portions of tissue to each of six dry BOD bottles. Each sample should weigh about 0.2 g. Add 4 ml of concentrated H_2SO_4 and 1 ml of concentrated HNO_3 to each bottle and place in a water bath at 58°C until the tissue is completely dissolved (30 to 60 minutes).

Cool the BOD bottles and add 0.0, 0.5, 1.0, 2.0, 5.0 and 10.0 ml aliquots of the working mercury solution containing 0.0 to 1.0 ug of mercury to the BOD bottles. Cool to 4°C in an ice bath and cautiously add 15 ml of potassium permanganate solution. Allow to stand overnight at room temperature under oxidizing conditions.

Add enough distilled water to bring the total volume to approximately 125 ml. Add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permangante.

Wait at least 30 seconds after the addition of hydroxylamine before proceeding. Treating each bottle individually, add 5 ml of the stannous sulfate solution and immediately attach the bottle to the aeration apparatus.

Continue with the procedure as given in Method 245.5 for soil CLP Inorganic SOW 785, Attachment 6, page D-55. Prepare a calibration curve by plotting the peak height versus the mercury concentration. The peak height of the blank is subtracted from each of the other values.

Sample Procedure

Weigh 0.2 to 0.3 g portions of the sample and place in the bottom of a dry BOD bottle. Care must be taken that none of the sample adheres to the side of the bottle. Add 4 ml of concentrated H_2SO_4 and 1 ml of concentrated HNO_3 to each bottle and place in a water bath maintained at 58°C until the tissue is completely dissolved (30 to 60 minutes).

Cool to 4°C in an ice bath and cautiously add 5 ml of potassium permanganate solution in 1 ml increments. Add an additional 10 ml or more of permanganate as necessary, to maintain oxidizing conditions. Allow to stand overnight at room temperature. Add enough distilled water to bring the total volume to approximately 125 ml. Add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

As an alternate to the overnight digestion, the solubilization of the tissue may be carried out in a water bath at 80°C for 30 min. Cool the sample and add 15 ml of potassium permanganate solution cautiously. Return the sample to the water bath and digest for an additional 90 min at 30°C (9). If this method is followed, the calibration standards must also be treated in this manner.

Calculation

Measure the peak height of the unknown from the chart and read the mercury value from the standard curve.

Calculate the mercury concentration in the sample by using the following formula:

$$\text{ug Hg/gram} = \frac{\text{ug Hg in aliquot}}{\text{wt. of aliquot in grams}}$$

Report mercury concentrations as follows:

Below 0.1 ug/gm, <0.1 ug; between 0.1 and 1 ug/gm, to nearest 0.01 ug; between 1 and 10 ug/gm, to nearest 0.1 ug; above 10 ug/gm, to nearest ug.

Quality Assurance

Standard quality assurance protocols should be employed, including blanks, duplicates, and spiked samples as described in the "Analytical Quality Control Handbook" (4). Spikes and

duplicates will be at the frequency/levels specified in the CLP Inorganic SOW 785. Control spikes for daily analysis will be the same level as matrix spikes.

Report all quality control data when reporting results of sample analyses.

Mercury spiking solution will be an organo-mercury compound (eg., methyl mercuric chloride) rather than inorganic mercury.

Precision and Accuracy

The following standard deviations on replicate fish samples were recorded at the indicated levels: 0.19 ug/gm $\pm$ 0.02, 0.74 ug/gm $\pm$ 0.05, and 2.1 ug/gm $\pm$ 0.06. The coefficients of variation at these levels were 11.9%, 7.0%, and 3.6%, respectively. Recovery of mercury at these levels, added as methyl mercuric chloride, was 112%, 93%, and 86%, respectively.

ATTACHMENT 3-4

METALS DIGESTION

Procedure

Mix the sample (ground tissue) thoroughly to achieve homogeneity. For each digestion procedure, weigh to the nearest 0.01 g, a 1.0 g portion of sample and transfer to a 100 ml teflon beaker equipped with a tight fitting lid. For tissue/fatty samples, always use $\leq$1.0 g. For a non-fatty matrix such as soil, a larger aliquot may be used.

Prepare a blank, a duplicate, and a spike for every five samples. Include a lab control blank, a control spike, and (if available) a control standard for every ten samples or sample fractions. Treat all of the audits in the same manner as the rest of the samples. (for definition of audit samples, see Table B).

Add 20 ml concentrated HNO_3, and 1 ml concentrated HF. Wearing gloves, mix the slurry and cover with a tight fitting lid. Heat the sample to 95°C and reflux to generate NO_x fumes. The brown NO_x fumes will escape though the beaker pour spout, or lift lid to check for fumes. Digest the covered sample overnight at 95°C.

Remove the lid and wash it and the sides of the beaker with Type II water (minimal amount) and 10 ml HCO_4. From this point on, do not leave sample unattended. Inexperienced analysts may want to wear a face shield. Work in a perchloric acid hood.

Raise the temperature to 160°C. Have a dropping bottle of concentrated HNO_3 near. If sample froths, add a drop or two of HNO_3. Repeat if necessary. Heat the samples until white fumes evolve and sample is near dryness. Fuming should coincide with the point where 1-2 ml remain in the beaker. Do not carry to dryness as the perchlorates may explode. Remove beaker from the heat while still moist. Sample may turn brown, but should not turn black or perchlorates may explode.

Wash down the sides of the beaker with several ml HNO_3 and Type II water. The sample is now ready for analysis per Inorganic SOW 785 Methods.

REFERENCES

1. Bishop, J.N., "Mercury in Fish", Ontario Water Resources Comm., Toronto, Ontario, Canada, 1971.

2. Boyle, H.W., et al., Adv. Chem. Serv., 60, 207 (1966).

3. Federal Register, Volume 41, No 232, p. 52780, Wednesday, December 1976.

4. Federal Register, Volume 44, No. 233, p. 69464, Monday, December 3, 1979.

5. Jones, J.W.; R. J. Gajan, K. W. Boyer; J. A. Fiorino; "Dry Ash - Voltammetric Determination of Cadmium, Copper, Lead, and Zinc in Foods." JOAC, 60, 825 (1977).

6. Jones, J.W.; R.J. Gajan; K. W. Boyer; J. A. Fiorino; "Dry Ash - Voltammetric Determination of Cadmium, Copper, Lead, and Zinc in Foods." JOAC, 60, 826. (1977).

7. Stalling, D.L.; R.C. Tindle; J.L. Johnson; "Cleanup of Pesticide and Polychlorinated Biphenyl Residues in Fish Extracts by Gel Permeation Chromatography." JOAC, 55; 32-38 (1972).

8. "Standard Methods for the Examination of Water and Wastewater", 14th edition (1975).

9. "EPA-CLP Inorganic Analysis: Multi-Media/Multi-Concentration", SOW No. 785, July 1985. Attachment 6: Method 245.5, Mercury in Sediments, pages D-52 through D-56. Annotated/Modified Method.

10. Handbook for Analytical Quality Control in Water and Wastewater Laboratories. U.S. Environmental Protection Agency, Technology Transfer, 1979.

11. "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", Physical and Chemical Methods Branch, EMSL-CI, revised October 1980. pages 27-29, 54. Primary Method: EPA 600/4-81-055. "EPA-CLP Inorganic Analysis: Multi/Media/Multi-Concentration", SOW No. 785, July 1985. Attachment #8: Method 335.2 (Sed.) CLP-M, Cyanide in Sediments, pages D-69 through D-80. Annotated/Modified Method.

12. "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268, 1978.

13. "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Technology Transfer. (1979).

REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS)

REVISED QUALITY ASSURANCE PROJECT PLANT

Project Title: Crab Orchard National Wildlife Refuge
EPA Project Officer: Richard Boice

Prepared by: O'Brien & Gere Engineers, Inc. Date: 11/19/86

Approved: _____ Date: 11/19/86
O'Brien & Gere Engineers, Inc.
Project Officer

Approved: _____ Date: 11/19/86
OBG Laboratories, Inc. Manager

Approved: _____ Date: 11/21/86
FWS Project Manager

Approved: _____ Date: 11/24/86
EPA Remedial Site Project Manager

Approved: _____ Date: 11/24/86
EPA QA Officer

CRAB ORCHARD NWR RI/FS
ADDENDUM NO. 2 TO QUALITY ASSURANCE PROJECT PLAN, REVISION 4
November 19, 1986

The following modifications and corrections to the Quality Assurance Project Plan (QAPP), Revision 4 for the Phase II sampling and analysis program.

1. SECTION 2

   a. Table 8A, page 1 of 1

      i) For item 5, Nitrosamines (low level, water), column 3 under ETC is changed from W/S to W.

      ii) For item 12, Special-Magnesium, column 4 under Rocky Mt is changed from W to W/S.

      iii) For item 21, Percent Solids is changed to include S under each of the four laboratory columns.

      iv) On the Addendum to Quality Assurance Plan, Revision 4, dated November 14, 1986, Section 2, item c. ii) is stricken. Extractions and analyses for CLP organics and nitrosamines will all be conducted by ETC.

2. SECTION 7, Table 10

   a. Pages 7, 9, 10, 20 and 21 of 25

      On the Spike Sample notation, SOW No. 784 (July 1984) is changed to SOW No. 785 (July 1985).

   b. Page 8 of 25

      The second to last notation is changed from "Furnace work will require duplicate analysis..." to "Furnace work will require spike analysis..."

CRAB ORCHARD NWR RI/FS
ADDENDUM TO QUALITY ASSURANCE PROJECT PLAN, REVISION 4
November 14, 1986

The following modifications and corrections to the Quality Assurance Project Plan (QAPP), Revision 4 for the Phase II sampling and analysis program in response to comments received on November 14, 1986 from the U.S. EPA on the QAPP, Revision 4.

1. SECTION 1

a. Table 2D, page 1 of 2

i) The list of compounds for CLP HSL Volatiles, number 35 should be deleted since total Xylenes was repeated twice (nos. 35 and 36).

ii) 4-nitrophenol is added to the list of compounds for CLP HSL Semi-volatiles.

b. Table 7, page 12 of 34

i) Small mammals for liver examination is included for Site #12.

ii) Six Phase I soil samples representing low, medium and high arsenic levels will be re-analyzed for arsenic.

c. Table 7, page 34 of 34

The total number of soil samples for arsenic is 10. The total number of analysis is 1587.

d. Table 7C

The total number of soil/sediment samples for arsenic is 11.

1. SECTION 2

a. Page 1 of 4

In Section 2.02, U.S. EPA's role in the Phase II sampling and analysis program is modified such that quality assurance review will be done by the Quality Assurance Office, U.S. EPA and data assessment will be done by the Contract Project Management Section, U.S. EPA.

b. Page 4 of 4

In Section 2.06, U.S. EPA's role in the Phase II sampling and analysis program is modified such that final data validation and data assessment will be done by the Contract Project Management Section, U.S. EPA.

c. Table 8A

i) Percent solids is scheduled for soil/sediment samples only.

ii) Due to holding time restrictions, samples for CLP organics will be extracted by O'Brien & Gere and sent to ETC for analysis.

3. SECTION 4

a. Page 3 of 20

In Section 4.03, the procedure for rinsing the stainless steel Kemmerer will be as described in Section 4.07.

b. Page 5 of 20

In Section 4.05, the split sampling procedure is scheduled for soil/sediment borings only. Surface soil/sediment samples will be collected using a shovel. Decontamination procedures are given in Section 4.07.

c. Page 5 of 20

A qualified O'Brien & Gere hydrogeologist will be present at the site during the installation of monitoring wells.

d. Page 6 of 20

i) Materials for monitoring well installation, including the PVC riser, will be washed and rinsed according to the procedures in Section 4.07.

ii) During the installation of each monitoring well, continuous split spoon (ASTM-D1586) samples will be collected and classified in the field by the hydrogeolist in accordance with the Unified Soil Classification System.

Grain size and Atterburg limits will be determined on samples at intervals selected by the hydrogeologist to confirm field classifications.

iii) All deep wells will be installed down to bed rock.

iv) At sites were only shallow wells are scheduled, the screens for the shallow wells will be located in the most permeable zone as determined by the field hydrogeolist.

At sites where both shallow and deep wells are scheduled in Phase II, the deep well will be first installed. The screen in shallow wells will in the most permeable zone based on the characterization of split spoon samples from the deep well.

e. Figure following page 6 of 20

The screen length will be 5 feet.

f. Table following page 17 of 20

i) The 1 liter glass sample container for low level nitrosamines (item no. 5) in water will be wrapped in aluminum foil.

ii) For low level PCB's in water (item no. 8), each sample will be collected in two 1 qt. glass containers for extraction per procedures in Attachment 7 of the QAPP.

4. SECTION 7, Table 10

a. Page 4 of 25

For nitrosamines in clean water samples, final extract volumes of 0.1 ml will be obtained to achieve an order of magnitude lower detection level than stated under Method 607. Visual observations will be used by laboratory personnel to determine if the samples are clean enough to achieve the lower detection level.

Quality assurance objectives are as given in page 4 of 25 for the low level nitrosamines, including deviations from the requirements under Method 607.

b. Page 6 of 25

For low level PCB's in water, general procedures are given in Attachment 5 and extraction procedures are given in Attachment 7.

Quality assurance objectives are as given in page 6 of 25 for the low level nitrosamines, including deviations from the requirements under Method 607.

c. Page 9 of 25

The detection limit are 0.2 ppb for mercury and 1 ppb for lead.

d. Page 10 of 25

Method reference for arsenic is 206.2. Detection limit for lead is 1 ppb.

e. Pages 11 and 22 of 25

Method reference for cyanide is 335.3

f. Page 18 of 25

For low level PCB's in water, general procedures are given in Attachment 5 and extraction procedures are given in Attachment 6.

CRAB ORCHARD NWR RI/FS
ADDENDUM TO QUALITY ASSURANCE PROJECT PLAN, REVISION 4
November 14, 1986

The following modifications and corrections to the Quality Assurance Project Plan (QAPP), Revision 4 for the Phase II sampling and analysis program in response to comments received on November 14, 1986 from the U.S. EPA on the QAPP, Revision 4.

1. SECTION 1

a. Table 2D, page 1 of 2

i) The list of compounds for CLP HSL Volatiles, number 35 should be deleted since total Xylenes was repeated twice (nos. 35 and 36).

ii) 4-nitrophenol is added to the list of compounds for CLP HSL Semi-volatiles.

b. Table 7, page 12 of 34

II reanalysis of 6 arsenic samples, CRT 7, 7C wt. adjusted. → I Small mammals for liver examination is included for Site #12.

2. SECTION 2

a. Page 1 of 4

In Section 2.02, U.S. EPA's role in the Phase II sampling and analysis program is modified such that quality assurance review will be done by the Quality Assurance Office, U.S. EPA and data assessment will be done by the Contract Project Management Section, U.S. EPA.

b. Page 4 of 4

In Section 2.06, U.S. EPA's role in the Phase II sampling and analysis program is modified such that final ~~data validation will be done by the Quality Assurance Office, U.S. EPA and~~ data assessment will be done by the Contract Project Management Section, U.S. EPA.

c. Table 8A

i) Percent solids is scheduled for soil/sediment samples only.

ii) Due to holding time restrictions, samples for CLP organics will be extracted by O'Brien & Gere and sent to ETC for analysis. → new

3. SECTION 4

a. Page 3 of 20

In Section 4.03, the procedure for rinsing the stainless steel Kemmerer will be as described in Section 4.07.

b. Page 5 of 20

In Section 4.05, the split sampling procedure is scheduled for soil/sediment borings only. Surface soil/sediment samples will be collected using a shovel. Decontamination procedures are given in Section 4.07.

c. Page 5 of 20

A hydrogeologist from O'Brien & Gere will be present at the site during the installation of monitoring wells.

d. Page 6 of 20

i) Materials for monitoring well installation, including the PVC riser, will be washed and rinsed according to the procedures in Section 4.07.

ii) During the installation of each monitoring well, continuous split spoon samples will be collected. Grain size and Atterburg limits will be determined on samples at regular intervals.

iii) All deep wells will be installed down to bed rock.

iv) At sites were only shallow wells are scheduled, the screens for the shallow wells will be located in the most permeable as determined by the field hydrogeolist.

At sites where both shallow and deep wells are scheduled in Phase II, the deep well will be first installed. The screen in shallow wells will in the most permeable zone based on the characterization of split spoon samples from the deep well.

e. Figure following page 6 of 20

→ The screen length will be 5 feet.

f. Table following page 17 of 20

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4. SECTION 7, Table 10

a. Page 4 of 25

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Quality assurance objectives are as given in page 4 of 25 for the low level nitrosamines, including deviations from the requirements under Method 607.

b. Page 6 of 25

For low level PCB's in water, general procedures are given in Attachment 5 and extraction procedures are given in Attachment 7.

Quality assurance objectives are as given in page 6 of 25 for the low level nitrosamines, including deviations from the requirements under Method 607.

c. Page 9 of 25

The detection ~~levels~~ limit are 0.2 for mercury and 1 ppb for lead.

d. Page 10 of 25

1ppb for Pb → Method reference for arsenic is 206.2.

e. Page 11 of 25

Method reference for cyanide is 335.3

f. Page 18 of 25

For low level PCB's in water, general procedures are given in Attachment 5 and extraction procedures are given in Attachment 6.

~~Page 22/25~~
CN 335.3

O'BRIEN & GERE

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O'BRIEN & GERE

December 19, 1986

Mr. Dick Ruelle
Resource Contaminants Assessment Coordinator
U.S. FISH AND WILDLIFE SERVICE
1830 Second Avenue
Rock Island, IL 61201

Re: Crab Orchard RI/FS
QAPP, Revision 4
Addendum No. 3

File: 3114.001

Dear Mr. Ruelle:

Enclosed is a copy of Addendum No. 3 to the Quality Assurance Project Plan (QAPP), Revision 4 for the Phase II sampling and analysis program.

This addendum replaces Attachment 3 of the QAPP and provides to be used for preparation, extraction, digestion and analysis of fish tissue. Analyses include pesticides/PCBs, mercury, cadmium and lead. The QA/QC audits and frequency are incorporated in the text. Control limits are based on the referenced Contract Laboratories Procedures.

The preparation and analysis will be subcontracted to Hazelton Laboratories, Madison, Wisconsin. They require 45 days from sample delivery to complete and submit data. It is desirable to get these analyses underway as soon as possible, so that the biota results may be included with the report of results for the other Phase II samples currently being analyzed.

O'Brien & Gere Engineers, Inc.
Box 4873 / 1304 Buckley Road / Syracuse, NY 13221 / (315) 451-4700
Blue Bell, PA / Boston, MA / Edison, NJ / Johnson City, NY / Landover, MD / New York, NY / St. Louis, MO

Please indicate your concurrence with these procedures by signing and returning a copy of this letter to me.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

Cornelius B. Murphy, Jr.

Cornelius B. Murphy, Jr., Ph.D.
Senior Vice President
Project Officer

SRG:dn/2:28
Enclosures
cc: Mr. Richard Boice (U.S. EPA) (5 copies)
Mr. John Hanson (Beveridge and Diamond)
Mr. Norrell Wallace (U.S. FWS)
Ms. Jean Sutton (U.S. DOI)
Mr. Bob Cowles (IEPA)
Dr. Dharmarajan R. Iyer
Mr. David R. Hill
Mr. Steven R. Garver

Approved _____ Date: _____
OBG Laboratories, Inc.

Approved _____ Date: _____
FWS Project Manager

Approved _____ Date: _____
EPA Remedial Site Project Manager

Approved _____ Date: _____
EPA QA Officer

O'BRIEN & GERE

November 14, 1986

Mr. Dick Ruelle
Resource Contaminants Assessment Coordinator
U.S. FISH AND WILDLIFE SERVICE
1830 Second Avenue
Rock Island, Illinois 61201

Re: Crab Orchard RI/FS
QAPP, Revision 4 Addendum
File: 3114.001

Dear Mr. Ruelle:

Enclosed is a copy of the Addendum to the Quality Assurance Project Plan (QAPP), Revision 4 for the Phase II sampling and analysis program. This Addendum to the QAPP (Revision 4) addresses comments received from USEPA subsequent to the submittal of the QAPP, Revision 4 and Phase II Site Operations Plan (SOP), Revision 4.

The comments were discussed in a conference call on November 14, 1986 between Dr. Dharmarajan Iyer and Mr. Rick Stromberg of O'Brien and Gere, Mr. Dick Ruelle of FWS, and Mr. Rich Boice of U.S. EPA. The attached Addendum includes modifications and corrections to the QAPP, Revision 4 that were agreed upon in the conference call. The U.S. EPA has agreed to approve the QAPP, Revision 4 subsequent to review of the Addendum.

Please contact me if you have any questions on the materials presented herein.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

Steven R. Garver, PE
Vice President

Encl.
cc: Mr. Richard Boice (U.S. EPA) (5 copies)
Mr. John Hanson (Beveridge and Diamond)
Mr. Norrell Wallace (U.S. FWS)
Ms. Jean Sutton (U.S. DOI)
Mr. Bob Cowles (IEPA)
Dr. Cornelius B. Murphy, Jr.
Dr. Dharmarajan R. Iyer
Mr. Dave R. Hill

O'BRIEN & GERE

November 11, 1986

Mr. Dick Ruelle
Resource Contaminants Assessment Coordinator
U.S. FISH AND WILDLIFE SERVICE
1830 Second Avenue
Rock Island, Illinois 61201

Re: Crab Orchard RI/FS
Quality Assurance Project
Plan, Revision 4
File: 3114.001

Dear Mr. Ruelle:

Enclosed is a copy of the Quality Assurance Project Plan (QAPP), Revision 4 for the Phase II sampling and analysis program. The Work Plan Supplement - Phase II Site Operations Plan (SOP), Revision 4 (November 1986) is provided separately. This QAPP (Revision 4) incorporates comments received from USEPA subsequent to the submittal of the QAPP (Revision 3) and Phase II SOP (Revision 3). The comments were discussed and modifications to the QAPP were finalized during a conference call between Mr. Steve Garver, Dr. Dharmarajan Iyer and Mr. Dave Hill of O'Brien and Gere, Mr. Dick Ruelle of FWS, and Mr. Rich Boice, Mr. Dave Payne and Dr. Chai Teng of U.S. EPA.

The Phase II sampling effort will be initiated on November 17, 1986, subject to approval of the QAPP and Phase II SOP. Please contact me if you have any questions on the materials presented herein.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

SRGarver

Steven R. Garver, PE
Vice President

SRG:wp
Encl.
cc: Mr. Richard Boice (U.S. EPA) (5 copies)
Mr. John Hanson (Beveridge and Diamond)
Mr. Norrell Wallace (U.S. FWS)
Ms. Jean Sutton (U.S. DOI)
Mr. Bob Cowles (IEPA)
Dr. Cornelius B. Murphy, Jr.
Dr. Dharmarajan Iyer
Mr. David R. Hill

REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS)

REVISED QUALITY ASSURANCE PROJECT PLANT

Project Title: Crab Orchard National Wildlife Refuge
EPA Project Officer: Richard Boice

Prepared by: O'Brien & Gere Engineers, Inc. Date: 11/10/86

Approved: _____ Date: 11/10/86
O'Brien & Gere Engineers, Inc.
Project Officer

Approved: _____ Date: 11/10/86
OBG Laboratories, Inc. Manager

Approved: _____ Date: _____
FWS Project Manager

Approved: _____ Date: _____
EPA Remedial Site Project Manager

Approved: _____ Date: _____
EPA QA Officer

QUALITY ASSURANCE PROJECT PLAN (QAPP)

REMEDIAL INVESTIGATION/
FEASIBILITY STUDY
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE
U.S. DEPARTMENT OF INTERIOR
MARION, ILLINOIS
AND
SANGAMO-WESTON, INC.
ATLANTA, GEORGIA

NOVEMBER, 1986

O'BRIEN & GERE ENGINEERS, INC.
1304 BUCKLEY ROAD
SYRACUSE, NEW YORK 13221

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REVISED QUALITY ASSURANCE PROJECT PLAN
CRAB ORCHARD NATIONAL WILDLIFE REFUGE
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

SECTION 1 - PROJECT DESCRIPTION

1.01 Introduction

O'Brien & Gere Engineers, Inc., is currently responsible for a Remedial Investigation/Feasibility Study (RI/FS) at the Crab Orchard National Wildlife Refuge in Marion Township, Williamson County, southern Illinois. Information obtained from previous studies and historic information provided by the Refuge Manager; lead to the selection of 31 sites (see Table 1 and Figure 1) within the Crab Orchard National Wildlife Refuge for investigation. Two additional sites were also included to serve as background or control sites.

In order to direct and control a multi-sited investigation representing various matrices (soil, sediment, water, biota, etc.) it was determined that the study be conducted in 2 phases.

The objective of Phase 1 was to identify, through a qualitative approach, whether contamination was present on a given site and to define the range of chemical compounds which contributed to the problem.

The Phase 1 sampling and analysis program was initiated in July 1985 and completed in November 1985. Phase 1 references and data are included in this QAPP for background information only, consequently they are not presented as items for EPA approval.

As required by the Environmental Protection Agency (EPA), a Quality Assurance Project Plan (QAPP) has been prepared for Phase II of this RI/FS and is presented herein.

At the conclusions of Phase 1 analytical results of the 500 plus samples were reviewed and 16 sites were selected for Phase 2 sampling. The selected Phase 2 sites will not receive a quantitative approach. The 16 selected sites will receive a comprehensive evaluation, horizontal and vertical, of the compounds identified in Phase 1. The information generated during Phase 2 will be used to complete the feasibility study.

This QAPP includes, in specific terms, the policies, organization, objectives, activities and specific Quality Assurance (QA) and Quality Control (QC) activities designed to achieve the data quality goals of this project. Where possible, existing QA/QC guidelines, policies, programs, etc., are incorporated into the QAPP by reference.

The purposes of this remedial investigation are: 1) to determine the nature and extent of any contaminant problem at several sites (Table 1) located around the eastern section of the Crab Orchard Lake (Figure 1) on the Crab Orchard National Wildlife Refuge and tributaries that drain into Crab Orchard Lake and 2) to gather all data necessary to support the Feasibility Study. This will involve the following activities:

- Determine current groundwater gradients where applicable.
- Determine the extent of groundwater contamination that has occurred and the rate and direction of contaminant migration.
- Assess levels of contaminated soil that may be present adjacent to disposal areas.
- Identify the areal extent of disposal areas.
- Identify specific contaminants which may pose acute or chronic hazards to public health, welfare or the environment.

- Identify pathways of contaminant migration from the sites.
- Define on-site physical features and facilities that could affect contaminant migration, containment, or cleanup.

O'Brien & Gere will furnish all personnel, materials and services necessary for or incidental to performing the remedial investigation on the Crab Orchard National Wildlife Refuge.

The remedial investigation consists of eight tasks:

Task 1 - Description of Current Situation

Task 2 - Investigation Support

Task 3 - Site Investigation

Task 4 - Preliminary Remedial Technologies

Task 5 - Site Investigations Analyses

Task 6 - Final Report

Task 7 - Community Relations

Task 9 - Additional Requirements

1.03 Site Location and History

Crab Orchard National Wildlife Refuge (CONWR or the Refuge) is located in southern Illinois primarily within Williamson County, but also extends into neighboring Jackson, Union and Johnson Counties. There are twelve lakes located within the Refuge including Crab Orchard Lake. Crab Orchard lake was completed in 1940 and has a surface area of 6,965 acres, a maximum depth of 30 feet and 635 acre-feet of storage capacity. The watershed drainage area is 109,261 acres. In addition to supporting an active sport fishing population, the lake serves as water supply (approx. 280,000 gallons per day) for the Refuge and

Federal Penitentiary located southeast of the Refuge. The City of Marion has a supplemental water intake in the Lake which has rarely been used.

The Refuge is administered by the U.S. Fish and Wildlife Service (FWS) of the Department of the Interior (DOI). During the early 1940s and continuing to the present, a number of industries have been active on the Refuge. Industrial activity was especially heavy during World War II when as many as 10,000 persons were employed by a number of defense-related industries. The section of the Refuge containing the industrial facilities lies within the eastern drainage area for the Crab Orchard Lake. The western portion of the lake has been used primarily for recreational purposes.

These industrial facilities were involved in a variety of manufacturing processes such as:

- Manufacture of land mines and bombs
- A munitions plant
- Manufacture of printing inks
- Production of radio speakers
- Metal plating, painting, metal work electrical work

To support these facilities, industrial dumps were developed within the Refuge. During the early 1940s, too, the Crab Orchard site was repeatedly sprayed with lead arsenate to control insects.

From the late 1970s through the present, sampling has been conducted to permit analysis of contamination. Until 1981, the main parameters of interest were lead, mercury, and other heavy metals, notably

cadmium. After 1981, analyses were conducted also for PCBs, dioxins, and benzo furans.

Phase I of the Remedial Investigation encompassed thirty-three (33) sites, including two control sites. The histories of each site are as follows:

Site 3: Area 11 South

Areas 11 and 12 are currently abandoned sites of explosives and nitrogen fertilizer manufacturing as well as munitions loading. The Olin Corporation is reported to have operated a dynamite line there which was later reportedly sold to U.S. Powder. A number of fires and explosions are known to have occurred in these areas. Use of lead azide in the area is suspected. RDX may have been used in this area. Many of the buildings and grounds have been "torched" to remove residuals of flammable material. Most of the buildings are covered with a spark-retarding asbestos siding material. Also, within Area 11 are storage areas where explosive powders were stored in rubber-lined underground trenches. A burning pad is evident to the south of Area 11 where oil residues, 50-calibre powder magazines and small powder cylinders are noticeable on the surface. The evaluations of these areas are not included in this scope of work.

The Area 11 South is located adjacent to what appears to be an old railroad bed. Much surface and buried litter is evident over an area of perhaps 10 acres. In addition to railroad track, ties and ballast, the following were also observed: cinders and charred wood, powder canisters, piping, metal, mesh, bricks, pumice blocks, 30- and

55-gal drums, reinforcing bars, a laboratory flask and miscellaneous wire and plastic articles. One mound on the bank just above the stream bed has several of what appeared to be metal vents on the top and a 4-in stainless steel pipe drain extending from the bottom. The stream bed west of the road appeared to contain especially heavy concentrations of debris. Black tars and ash were evident in the stream bed.

Site 4: Area 11 North

The Area 11 North appears to have been the site of a large (2 to 3 acre) impoundment. The impoundment is flat in the middle and has small intermittent stream or marsh areas bordering the east and west boundaries. Water appears to flow from south to north following periods of precipitation. The reinforced concrete remains of a dam can be seen at the northwest end of the site. A large earth bunker is located immediately to the west. It may have been built with earth excavated from the semi-marshy lagoon area and may have been constructed to protect the explosives processing areas located further to the west. It was suggested that RDX or magnesium may have been stored underwater here or the area may have been used to detonate explosives or for experimental detonations. The level bottom of the impoundment shows a number of bare patches of fine white silt or clay. Other weathered areas showed horizontal layering of white and gray sediments. A number of dynamite-type fuses were noticed here as well as a small powder carrier, 1.5-in dia by 3 in, with the fuse intact. Small lead chunks were also observed.

Site 5: Area 11 Acid Pond

The Area 11 Acid Pond is a diked impoundment approximately 300 ft x 150 ft which received drainage flowing north from the Area 11 process buildings. The dike extends 5 to 6 ft above the current water level. A 12 inch diameter pipe exits to the west through the levee to a valve box which controls the discharge from the pond to a small stream. This drainage then exits through the woods and swampy areas to the north. It is claimed that a spill of low-pH water (nitric acid) from the pond years ago killed all of the downstream vegetation for 1/4 mile. A large stand of dead trees is still visible along the creek north of the pond.

Sites 7, 8, 9, 10 and 11

D AREA SOUTHEAST DRAINAGE

D AREA SOUTHWEST DRAINAGE

P AREA NORTHWEST DRAINAGE

WATERWORKS NORTH DRAINAGE

P AREA SOUTHEAST DRAINAGE

The Olin D and P Areas are active Olin operations north of Crab Orchard Lake. Explosives are currently manufactured in the D Area while research and development is conducted in the P Area. It is likely that chemicals handled in the P Area are non-conventional or "exotic". Universal Match also previously conducted operations here under contract to the DOD. Their operations ceased after a large explosion.

Sites 7, 8, 9, 10 and 11 are locations within various drainage channels leading from the Olin D and P Areas. These discharge to the Lake near the Refuge Waterworks.

Site 7A:D Area North Lawn

There is a large (about 3 acre) lawn located northwest of the active Olin D Area complex. It is claimed that barrels of chemicals were dumped on a knoll within this lawn. No evidence of a knoll was seen during the site visit, but a number (about 8) of depressed brown patches were evident on the lawn. A visually clean drainage channel is located south of the lawn and exits under the fence to the west. Other moist drainage areas extend to the wooded area to the west of the site.

Site 11A: P Area North

Located outside of the fence north of the Olin P Area is an abandoned L-shaped loading area with connecting covered walkways approximately 100 ft and 85 ft. The central structure contains a loading dock and a steamhouse containing a concrete pit with about 5 ft of clear standing water. An old roadbed runs west and north of the structure and draining swales surround all of the buildings. An abandoned (?) sewer line also runs across the north edge of the site. It has been reported that contaminants were dumped on the ground outside of the building.

Site 12: Area 14 Impoundment

Area 14 was a site of munitions loading activity. Many of the buildings have been abandoned or demolished, but a few industries presently occupy some of the buildings. Historic aerial photos indicated what appeared to be landfill activity in the field east of the presently-occupied buildings. During the site visit the remains of a 100-ft dia circular impoundment were found at this site. The interior of the impoundment is presently overgrown with trees with trunk diameters of 8 to 10 in, indicating the date of the impoundment closure at about 1955 to 1965. The impoundment walls are about 6 ft high and the north wall has been breached to allow drainage to flow from the impoundment to an adjoining field. Several black oily pools are evident within and outside the basin. Other bare patches of black sediment and tars are located around the basin floor.

Site 13: Area 14 Change House Site

Southeast of the active Diagraph-Bradley buildings on Area 14 was an old building which was recently demolished. Formerly, it was the site of a "Change House" where workers changed their clothing after working in the adjacent bomb-loading buildings. At one time a company named CTI (Chemicals and Technology, Inc.??) manufactured explosives and other chemicals in this building. Other industries may also have occupied this building. The change building was supposedly located across from the bomb-loading building on a plot of land just southeast of the intersection of two roads on the north edge of a big dirt mound. The concrete floor of the change house is under this mound. Aerial photos show another building (no longer present) further east of the

corner; field inspection revealed several 1/2-in reinforcing rods imbedded in concrete near the corners of this building.

Site 14: Area 14 Solvent Storage

Diagraph-Bradley or Diagraph Marking Systems currently operates within a complex of buildings in Area 14. They produce inks, stencils, stencilboards and marking pens. Linseed oil and various solvents are handled in bulk and in drums here. Some of the bulk solvents noted were: T25 Xylene, T8 Diacetone Alcohol, T9 Diethylene Glycol, and T18 Methyl Cellosolve. Several compressed gas cylinders are also present. At least two drum storage areas containing 50 to 200 drums were also noted. Spill containment facilities are minimal. A drainage ditch runs north parallel to the road west of the buildings. Process water from the Diagraph-Bradley buildings enters this ditch from a standpipe.

Sites 15 and 16

AREA 7 PLATING POND

AREA 7 INDUSTRIAL SITE

Area 7 contains a complex of 33 identical buildings which have been used for a variety of industrial purposes during the past 40 years. Each of the six rows of buildings was previously served by a railroad siding.

Within a wooded rise to the south is located a small pond (approximately 50 ft x 30 ft) which is bermed about five ft above the current water level. The current water depth is estimated to be about four ft.

It is claimed that this pond was used to receive plating wastewaters from Olin operations which were located in this area at one time. PCBs, lead and other heavy metals may be of concern here.

Many of the buildings on the Area 7 site are used for dry warehousing purposes. However, two specific locations have been specified for sampling. Buildings 3-4, 3-5, and 4-4 are used by Pennzoil for waste oil recovery and recycling operations. Black residues are noticeable around some of these buildings. Buildings 5-2 and 5-3 are used by a refurbisher of mining machinery. Black residues are also evident around these buildings. A drainage channel runs from south to north through the center of the site.

Site 17: Job Corps Landfill

Northeast of the Refuge Waterworks is a small (approximately 10 acre) pond created by Job Corps workers in the mid-1960's. Attention has recently been brought to this pond because as many as thirty or more geese carcasses have been found floating on the water or littering the shores. Some of these carcasses have been relatively fresh while others were in various state of decay. The Fish and Wildlife Service has completed extensive analyses of these carcasses and has ruled out a variety of potential chemical causes. A definite conclusion has not yet been reached.

The "Job Corps" landfill was discovered while investigating the geese kills. It is located within a wooded area to the north and adjoining the pond and covers an area of perhaps an acre of more. It appears to be mainly surface litter dumped in spots and perhaps spread

around, although deeper spots cannot be ruled out. Many of the surface articles appear to be connected with food preparation, e.g. institutional-size food cans, and a variety of bottles. The bottle styles and labels suggest a date of the mid-1950's, which was consistent with a 1956 Illinois automobile license plate also found. Many of the debris piles are overgrown by thick brush. Two bare patches (less than 6-ft diameter each) were located among the debris. Mica flakes and small electrical contacts were found in one of these. It is claimed that small electrical capacitors were also found here, but none were noted during this site visit. Probing with a trowel revealed no further debris beneath the top inch of soil.

Site 18: Area 13 Loading Platform

On the northwest end of the Area 13 munitions storage bunkers is a concrete loading platform adjacent to the abandoned and dismantled rail line. It is reported that munitions-type chemicals were dumped off the platform. The site inspection indicated that the elevated concrete loading dock is about 235 ft long by 10 ft wide and about 5 ft high. The dock is supported on concrete posts spaced 9 ft apart. The northwest side contains stone bedding (probably from the oil railroad bed) with a number of small areas of ponded water. No unusual vegetation changes were detected. The only unusual item was a pile of dirt and stone rubble off the west end of the dock with a rusted drum shell nearby.

Site 19: Area 13 Bunker 1-3

Area 13 contains approximately 85 bunkers which were originally built for storage of 500-lb bombs. Most of them still contain explosives, leased mainly to Olin and U.S. Powder. Agricultural fields are cultivated between the bunkers. Formerly, they were fruit orchards.

It has been reported that chemicals were poured out near Bunker 1-3, probably in the field next to it. A site inspection did not reveal any significant signs of impact. Evidence of fill activity (scattered red bricks) is widespread. An L-shaped area of brown vegetation difference was noted to the west side of the bunker.

Site 20: D Area South

An abandoned building is located within the fenced southeastern end of the Olin D Complex. It was reported that chemicals were dumped here. A drainage swale originating at the building runs east outside of the fence. A four-in pipe (dripping) extends from the Olin Area under the fence and discharges to this ditch. A slight sheen was noticeable on the surface water in pooled areas of the ditch.

Site 21: Southeast Corner Field

At the southeast corner of the refuge is a field which is thought to be the site of a very old landfill. A pile of concrete pieces, possibly from an old bridge, is located immediately inside the fence. The topography gradually slopes to the south and east with a swampy drainage ditch at the bottom of the slope. No other evidence of debris could be found. Trees as large as 24-in in diameter suggest that the

area has not seen any soil-disturbing activity within the past 60 to 70 years.

Site 22: Old Refuge Shop

North of the refuge along Wolf Creek Road is the old refuge headquarters, now leased by Diagraph Bradley. Behind this building is located the old shop area of the refuge. Pine poles were treated here with pentachlorphenol and shipped to various spots around the country. Outside the fence to the north is a small pool which receives drainage from the old shop area. The pool contains a green-yellow scum and drains through the woods to the northwest.

Site 24: Pepsi-West

The Pepsi Cola Bottling Company in Marion could potentially discharge to Crab Orchard Creek. It is not known whether the City or State monitor environmental activities here. A site inspection indicated that it was unlikely that discharges issued directly south to the Creek, since the entire south end of the property rises 4 to 8 ft in elevation above the parking lot. Drainage ditches, however, were located to the north adjacent to the street. These probably receive surface runoff only.

Site 25: Crab Orchard Creek at Marion Landfill

The old Marion landfill is off Old Creal Springs Road and directly abuts Crab Orchard Creek. It has apparently been inactive for a number of years. A visible face of trash can be seen by travelling

upstream several hundred yards from the road. Near to this is a small pond (approximately 3/4 acre).

Sites 26 and 27:

CRAB ORCHARD CREEK BELOW MARION STP

CRAB ORCHARD CREEK BELOW 157 DREDGE AREA

The Marion sewage treatment plant discharges to Crab Orchard Creek somewhere upstream of Court Street. A number of samples downstream from the Marion STP are scheduled to assess the quality of various stretches of Crab Orchard Creek.

Site 28: Water Tower Landfill

Aerial photos indicate landfilling activities adjacent to the water tower near Areas 7 and 14. These activities are not visually apparent today. The sloping face northeast of the water tower is heavily overgrown with briars and rutted with several major gullies. Only a small amount of refuse is evident on this slope. A previous soil sample taken in this area showed 800 ppm lead concentration. More activity is evident in the woods at the bottom of the slope. A number of rusted drums, metal parts and tar residues can be found here. Standing water in the main drainage gully shows a slight sheen on the surface. Several small mounds are within the woods and a larger mound is located at the top of the hill.

Site 29: Fire Station Landfill

Located southwest of the refuge fire station is a large field which was used for storage of mining machinery until several years ago. The northern and western edges of this field show evidence of a large dump site. Debris is evident on the face which drops 4-5 ft. to a swampy area to the west. Previous sampling near an evergreen tree on the north side showed lead concentrations of 553 ppm. A slight sheen is noted in spots within the swamp. Most of the debris consists of concrete, metal, wire and other machinery-related items. It was reported that Olin dumped heavily here and there once was a very hot fire. Ignitable magnesium is suspected to be in the fill. An empty 30-gal drum labelled "Magnesium Powder" was found along the south portion of the eastern face.

Site 30: Munition Control Site

A munition control site is established on an area where the operations involved only ammunitions manufacture.

Site 31: Refuge Control Site

A control sampling station is established on an uncontaminated area of the refuge behind the new Refuge headquarters. Selection of the control site was coordinated with the Refuge Manager, following a site visit.

Site 32: Area 9 Landfill

The Area 9 Landfill was used during the 1950's and early sixties and was probably closed in 1964. The Landfill is located below

approximately 100 yds south of Crab Orchard Lake and approximately 100 yards east of the building complex. Runoff can drain from the landfill into an intermittent creek and then to the Lake. The limits of the landfill are discernible by changes in the topography and vegetation. It is approximately 2.5 acres with a fill thickness of 8 to 10 feet in the middle and 6 feet at the edges. Waste materials are exposed at locations where cover material has eroded. Some areas are void of vegetation.

The volume of the landfill is estimated to be from 16,000 to 35,000 cubic yards. Materials visible on the surface appear to be electrical components consisting of small capacitors, capacitor parts, large chunks of a golden resin, and a large number of 3-inch steel cuplike pieces.

Wastes were burned, compacted in a swale and covered when the landfill was active. Specific compounds of concern include lead, acetate, PCBs (Aroclor 1254 and 1242), and PCB burning products. Other possible materials from capacitor manufacturing include mica, silver, cyanide, aluminum hydroxide, aluminum oxide, gold, copper, zinc, hydrochloric acid, styrene, nitric acid, phosphoric acid, and borates. Other industrial wastes may include cyanides, printing inks and lead-based explosives. A magnetometer survey indicated a high concentration of metals on the east side of the landfill.

Site 33: Area 9 Building Complex

The Area 9 Building Complex was leased during the period from 1946 to 1962 as the Ordill Facility containing the Sangamo Capacitor Division. Manufacturing operations began in the early 1950's. This

division manufactured power factor capacitors, AC motor run capacitors, and a variety of DC capacitors. The components were of various types and included aluminum, electrolytes, mica, and silver and lead foil. The Division also manufactured small transformers that used mineral oil as a dielectric.

Subsequently, Olin Corporation started using the industrial facilities at the site. Olin manufactured explosives that were used to start jet engines. The company used nitro-glycerine in its operation.

Site 34: Crab Orchard Lake

Crab Orchard Lake (completed in 1940) has a surface area of 6,965 acres, a maximum depth of 30 feet, and 635 acre-feet of storage capacity. The watershed drainage area is 109,261 acres. The lake has a retention time of approximately 0.8 years. Water enters the lake through several creeks, including Crab Orchard Creek on the eastern end of the lake and an intermittent creek adjacent to the Area-9 Landfill. Water leaves the lake through Crab Orchard Creek on the western end of the lake. In addition, 280,000 gallons/day of water is used by the Refuge.

The eastern section of the lake is near several manufacturing operations established since the 1940s.

1.03 Project Objectives

The primary objectives of the RI/FS are to determine any hazards to human health and the environment as well as to recommend the most cost-effective source control and off-site remedial actions. Source

control remedial actions include measure to prevent, reduce, or eliminate contamination either by containing the hazardous wastes in place or removing them from the site. Off-site remedial actions include measures to mitigate the effects of hazardous waste contamination that has migrated beyond the site. Appropriate source control and off-site remedial actions will be formulated and analyzed in detail after sufficient data have been generated through the remedial investigation.

Based upon existing data, remedial actions that may be appropriate for the CONWR site include, but are not limited to, one or a combination of the following:

- No action.
- Removal and disposal of waste material.
- Solidification or stabilization of waste material.
- In place reconstruction or encapsulation of waste material.
- Contaminated soil incineration.
- Continued off-site monitoring.
- Limit access to contaminated areas.
- Groundwater collection and treatment systems.
- Surface water drainage measures to prevent ponding on or near sites of contamination.
- Construction of groundwater barriers.
- Construction of a clay or synthetic cap over contaminated.

Presently, the available data and information on the site are insufficient to allow a definitive selection, screening, and feasibility study of remedial action alternative.

1.04 Project Description

The remedial investigation/feasibility study (RI/FS) for the Crab Orchard National Wildlife Refuge Site is intended to determine the nature and extent of contamination, to develop and evaluate remedial alternatives and to identify cost-effective remedial actions to be taken at contaminated sites on the refuge which reduce risks to acceptable levels. To accomplish this, the following tasks will be completed:

- characterize the on-site soil, sediment, water and biological samples for the presence of hazardous contaminants (includes landfill, surface soil, pond and lake water).
- identify pathways of chemical migration from the site.
- characterize the off-site soil, sediment, water and biological samples for key hazardous components.
- determine and describe on-site physical features that could affect migration of key hazardous components, methods of containment, or methods of remedial action clean-up.
- develop viable remedial action alternatives.
- permit the evaluation of the remedial action alternatives.
- recommend the most cost-effective technically feasible remedial option which has the ability to reduce impacts on human health, welfare and the environment to an acceptable level.
- prepare a conceptual design of the recommended remedial action alternative.

TASK 1 - DESCRIPTION OF CURRENT SITUATION

O'Brien & Gere has obtained available background information pertinent to the sites. The data gathered during any previous investigations or inspections and other relevant data were used in developing the RI work plans. A partial list of sources on published and unpublished data available on Crab Orchard Creek watershed and Crab Orchard Lake is included in the Work Plan Supplement (December 1985).

The sub-tasks include site background, nature and extent of the problem at the sites under investigation and a history of response actions.

TASK 2 - REMEDIAL INVESTIGATION SUPPORT

Prior to initiating the Phase I field investigations, the following preliminary work was completed.

A. Site Visit

Initial site visits were conducted to become familiar with site topography, access routes, and proximity of receptors to possible contamination, and to collect data to support the Site Health and Safety Plan. Site surveys were conducted to identify and stake boundaries of known contaminated areas, monitoring wells, and soil borings, and to identify sediment sample locations for Phase I sampling and analysis. The visit was used to verify the site information developed in Task 1. The Site Health and Safety Plan was amended as a result of this visit.

B. Site Maps

As part of the Remedial Investigation Report, O'Brien & Gere will prepare site maps showing all wetlands, water features, drainage patterns, tanks, buildings, utilities, paved areas, easements, right-of-ways, and other features. The site maps and all topographic surveys will be of sufficient detail and accuracy to locate and report all existing and future work performed at the sites. Areas of investigation will be mapped using existing topographic maps or aerial photos. After the analytical data have been reviewed and where necessary for remedial efforts, the topographic maps will be prepared with 1-foot contours referenced to the National Geodetic Vertical Datum with a scale of 1 inch to 50 feet. The maps will extend 200 feet beyond site boundaries and include all drainages to Crab Orchard Lake.

Boundary lines encompassing contaminated areas will be identified. The boundary lines for the landfill study sites will be identified using results from magnetometer and electromagnetic measurements. The boundary conditions will be set so that subsequent investigations will cover the contaminated media in sufficient detail to support the feasibility study. The boundary conditions may also be used to identify boundaries for site access control and site security. If necessary, a fence or other security measures may be installed as an initial remedial measure.

C. Dispose of On-Site Generated Waste

All wastes generated by on-site activities will be labelled, drummed and stored within controlled-access areas. Wastes which will be drummed include: all drill cuttings, all purged groundwater from well development, decontamination wash water and disposable protective clothing. This practice was followed during Phase I investigations. These materials, if contaminated, will be properly disposed of during cleanup actions as identified by the feasibility study.

TASK 3 - SITE INVESTIGATIONS

The remedial investigations include components necessary to characterize the site and its actual or potential hazard to public health and the environment. The site investigations will generate data of adequate technical content to support detailed evaluations of alternatives during the feasibility studies.

The sites listed in Table 1 fall under five categories.

1. Landfills
2. Surficial Contaminant Sites
3. Streams
4. Ponds
5. Lake

The sub-tasks under site investigations include:

A. Geophysical Surveys

B. Hydrogeologic Investigations

C. Groundwater Sampling and Analysis

D. Soil Investigation

E. Surface Water and Sediment Sampling and Analysis

F. Fish Sampling and Analysis

The site investigations include two phases. Phase I, which was completed in November 1985, included geophysical surveys, hydrogeologic investigations, installation of groundwater monitoring wells, and a screening of each site to analyze composited samples for a broad array of potential contaminants as listed in Table 2A. Selected samples were confirmed by a full analysis for priority pollutants.

Phase II, for which this QAPP has been developed, consists of the additional sampling and analysis to fill in data gaps identified in Phase I and further assess the extent of contamination at sites where materials of concern are found. Analytical parameters included for Phase II Site Investigations are listed in Table 2B, with reference to Table 2D for a list of compounds within each of these parameters.

A. Geophysical Surveys

Geophysical investigations were conducted in Phase I to determine the extent of soil and groundwater contamination, if any, in the vicinity of several specified study sites. In particular, the geophysical investigations were conducted at areas of suspected landfill activities, and consisted of magnetometer and electromagnetic induction (EM) surveys.

B. Hydrogeologic Investigations

The results from hydrogeologic investigation will be used to determine the present and potential extent of groundwater contamination, if any, and to evaluate the suitability of the site for on-site waste containment. Efforts began with a

survey of previous hydrogeologic studies and other existing data (completed as part of Task 1 a and c). The survey along with additional work in this investigation will address the degree of hazard, the mobility of chemicals considered, the soil attenuation capacity and mechanisms, discharge/recharge areas, regional flow direction and quality, and effects of any pumping alternative. Sampling programs for this Remedial Investigation has been developed to determine the horizontal and to vertical distribution of chemicals considered and predict the long-term disposition of such chemicals.

C. Sampling and Analyses of Groundwater

Nine groundwater monitoring wells were installed during the Phase I field effort. Additional monitoring wells will be installed during Phase II. All the monitoring wells will be sampled and the water analyzed for contaminants of concern.

Then, based on the geophysical results (Task 3a) and results of contaminant analyses, the extent and scope of any additional hydrogeologic investigation will be determined.

D. Soil Investigation

The two phased investigation program was developed to identify the location and extent of surface and subsurface soil, and sediment contamination. This process overlaps with certain aspects of the hydrogeologic study, e.g.,

characteristics of soil strata are relevant to both the transport of contaminants by groundwater and to the location of contaminants in the soil. Several soil samples and soil borings were collected for analysis from various sampling sites around the refuge during Phase I. Additional samples are scheduled to be collected in Phase II.

E. Surface Water and Sediment Investigation

O'Brien & Gere has developed and conducted a Phase I program to determine the overall extent of any water and sediment contamination on selected refuge lakes, marshes, ponds and streams. The initial process will overlap with the investigations scheduled in Phase II.

F. Fish and Wildlife Investigations

Selected species of fish have been collected, during Phase I, from Crab Orchard Lake by the FWS.

Table 7A lists the species and number of fish per site. Fish samples will be filleted (see Attachment 3) and analyzed for residual levels of contaminants previously identified in landfills and other contaminated areas on the refuge. Skin-on fillets will be used for carp and bass and skin-off fillets for catfish and bullhead. Additionally, percent lipids will be determined on the fish portions selected for analyses.

TASK 4 - PRELIMINARY REMEDIAL TECHNOLOGIES

A. Post-Investigation Evaluation

Either during or following the site investigations, O'Brien & Gere will assess the investigation results and recommend preliminary remedial technologies best suited to specific contaminant problems for each site. They will provide the basis for developing detailed alternatives needed for the completion of the feasibility studies. The data generated during the remedial investigations will generally be limited to accomplish the following:

1. Recommend types of remedial technologies appropriate to physical and site contaminant conditions.
2. Recommending whether or not to remove some or all of the waste for off-site treatment, storage, or disposal.
3. Determine the compatibility of groups of wastes with other wastes and with materials considered as part of potential remedial action. Recommend alternatives for treatment, storage, or disposal for each category of compatible waste.

TASK 5 - SITE INVESTIGATIONS ANALYSIS

The results of Tasks 1 through 4 will be used to prepare a thorough analysis and summary of all site investigations. The objective of this task is to ensure that the investigation data are sufficient in quality and quantity to support the feasibility studies.

The results and data from all site investigations will be organized and presented logically. The geographic groupings listed on Table 1

will form the basic structure for all of the assessments. This will permit the assessment of transport modes and impact to receptors.

A. Data Analysis and Endangerment Assessment

The site investigation data will be analyzed to develop a summary of the type and extent of contamination at the sites. The summary will describe the quantities and concentrations of specific chemicals at each site and ambient levels surrounding the sites. Ambient samples will be collected from control sites.

Data collected during the RI phase will also be evaluated to determine if environmental conditions or materials at the site present potential hazards to human health or welfare, or to the environment. Existing standards will be reviewed to help formulate conclusions and recommendations regarding the hazard potential of the site. If additional hazards are identified, the risks associated with each hazard will be summarized.

This analysis will discuss the degree to which either source control or off-site measures are required to significantly eliminate the threat, if any, to public health or the environment. If the results of the investigation indicate that no threat or potential threat exists, a recommendation of no remedial response will be made.

A technical memorandum will be prepared by the Respondents summarizing the hazard evaluation process and presenting the results of the hazard assessment.

TASK 6 - FINAL REPORT

A final RI report will be prepared to consolidate and summarize the data collected during the RI. The report will include a discussion of the data acquired during the RI and the hazard identification and risk potential of the contaminants detected. Ten copies of the remedial investigation report will be submitted to the FWS. The report will be structured to enable the reader to cross-reference with ease.

TASK 7 - COMMUNITY RELATIONS

The Community Relations program is included as Task 7; however, the dissemination of information to the public will be coordinated by the FWS throughout the duration of the study. O'Brien & Gere will provide personnel, at the Service's discretion, to support the programs as *community relations must be integrated closely for all remedial response* activities.

The objectives of this effort are (1) to keep the community informed as to the study progress, (2) to achieve community understanding of the actions taken, and (3) to obtain community input, and support prior to selection of the remedial alternative(s).

TASK 8 - ADDITIONAL REQUIREMENTS

A. Reporting Requirements

O'Brien & Gere will prepare monthly reports to describe the technical and financial progress of the project. These reports will discuss the following items:

1. Identification of sites on which activity took place and the nature of those activities.
2. Status of work at the site and programs to date.
3. Percentage of completion.
4. Difficulties encountered during the reporting periods.
5. Actions being taken to rectify problems.
6. Activities planned for the next month.
7. Changes in personnel
8. A comparison of target and actual completion dates for each element of activity including project completion and an explanation of any schedule deviations in the work plan.
9. Progress Reports on Items 1 through 8 will be submitted to FWS, who shall in turn relay them to USEPA and IEPA.
10. A Work Plan that includes a detailed technical approach and schedules will be submitted for the proposed feasibility study.

B. Site Health and Safety Plan

Prior to conducting any field activities O'Brien & Gere will provide any necessary modifications to the Site Health and Safety Plan as presented in Appendix C of the Work Plan dated June 1985. The plan is consistent with:

Section 111(c)(6) of CERCLA.

EPA Order 1440.3 - Respirator Protection

EPA Order 1440.2 - Health and safety requirements for employees engaged in field activities.

EPA Occupational Health and Safety Manual.

Other EPA guidance as provided.

State Safety and health statutes.

Site conditions.

EPA Interim Standard Operating Safety Guide (September 1982) and applicable OSHA standards.

C. Quality Assurance/Quality Control (QA/QC)

O'Brien & Gere has prepared a Quality Assurance Project Plan (QAPP) for the sampling, analysis, and data handling aspects of the remedial investigation which is presented in Appendix A of the Work Plan dated June 1985. The QAPP plan is consistent with U.S. Fish and Wildlife Service, State and Federal EPA requirements. The plan addresses the following points:

1. QA Objectives for Measurement Data, in terms of precision, accuracy, completeness, representativeness and comparability.
2. Sampling Procedures.
3. Sample Custody.
4. Field Equipment, Calibration Procedures, References and Frequency.
5. Internal QC Checks and Frequency.

6. QA Performance Audits, System Audits, and Frequency.
7. QA Reports to Management.
8. Preventative Maintenance Procedures and Schedule.
9. Specific Procedures to be used to routinely assess data precision, representativeness, comparability, accuracy, and completeness of specific measurement parameters involved. This section will be required for all QA project plans.
10. Corrective Action.

D. Site Sampling Plan

Site specific sampling plans for Phases I and II of site investigations have been developed for this Remedial Investigation, and are summarized in Section 1.05 of this QAPP. The sampling plan covers the sampling efforts described in the Remedial Investigation work plan and addresses the following topics:

- Sample types and tentative locations
- Sample equipment and procedures
- Sample handling, custody procedures, and preservation
- Sample documentation
- Sample shipping
- Analytical arrangements (scheduling)
- Analytical procedures
- QA/QC review procedures of data
- Analytical review of data

- Disposal of unused samples

1.05 Sampling and Analysis

Phase I sampling and analysis have been completed and details are set forth in Appendix B of the Site Sampling Plan dated 6/85. Additional details are included in the Work Plan Supplement dated 12/85. The Phase I sampling results and Phase II sampling program are presented in detail in the Work Plan Supplement, Phase II Site Operations Plan (Revision 3) dated 10/86.

Sampling activities under various Remedial Investigation Tasks are shown in Table 4. A listing of individual samples scheduled and rationale for Phases I and II sampling and analysis are included in the Work Plan Supplement - Phase II Site Operations Plan, September 1986. For Phase I, a summary of the analysis sets and sampling and analysis by sites, analysis sets and sample types are presented in Table 5. For Phase II, detailed sampling and analysis by site, parameters and sample types are presented in Table 7 and a summary in Table 6.

1.06 Project Schedule

The proposed project schedule is illustrated in Figure 2. This schedule was developed for planning purposes. Several tasks identified in the Work Plan emphasize uncertainties or contingent items which may be defined at a later date depending on the results of analytical data or engineering assessments. Therefore, schedule modifications may be necessary as these tasks are encountered. Phase II will be initiated upon approval of the QAPP and Site Operations Plan. Upon approval of

the QAPP and Site Operations Plan, a detailed time table will be provided identifying tasks completed and tasks projected to be completed.

SECTION 2 - PROJECT ORGANIZATION AND RESPONSIBILITY

2.01 Functional Activities

Table 8 lists the functional activities of this project and the firms responsible for the particular activity. Analytical laboratories scheduled to perform analysis on Phase II samples for the parameters (in Tables 2B and 7) are identified in Table 8A.

2.02 Project Organization

Table 9 lists the primary contacts for the project. Project technical personnel and quality assurance personnel are indicated in the project organization chart (Figures 3 and 4 respectively). Primary responsibility for project review rests in the NWR Resource Contaminants Assessment Coordinator.

Additionally, the USEPA will serve in a review capacity on issues that relate to human health, while the FWS functions in a equal capacity on issues that impact on wildlife. The USEPA will also supply performance evaluation samples and conduct performance and system audits.

The USEPA On-scene Coordinator will provide an interpretive review and oversight during the course of the RI/FS. Quality assurance review and data assessment will be conducted by O'Brien & Gere, USEPA Quality Assurance Officer and Columbia National Fisheries. CPMS

2.03 Project Manager (O'Brien & Gere)

The Project Manager, shown in Figure 3, will have primary responsibility for overseeing all facets of the project on a day-to-day basis. Specifically, his duties will include:

- Project scheduling
- Budget control
- Subcontractor performance review
- Review of interim reports
- Responsible for project coordination and communication
- Project deliverables
- Responsible for establishing a project specific record keeping system
- Project close-out

To accomplish the wide range of analysis, it is intended to utilize the services of four laboratories. The following list identifies the laboratories, and Table 8A identifies analysis to be performed by these laboratories.

OBG Laboratories, Inc. - Syracuse, New York

Environmental Testing and Certification (ETC) - Edison, New York

Ray F. Weston - Westchester, PA

Rocky Mountain Analytical Laboratory - Wilson Laboratories

2.04 Quality Assurance Officer

The Quality Assurance (QA) Officer shown in Figure 3 is responsible for the monitoring and supervision of the QA/QC program. The QA Officer reports directly to the Project Manager and his responsibilities include:

- Insure field personnel are both familiar with and adhering to proper sampling procedures, field measurements sample identification and chain-of-custody procedures.

- Contact the laboratory to insure that samples received by them have been properly identified and packaged.
- Monitor and audit the performance of the QA procedures.
- Conduct field and office audits.
- Insure that USEPA performance audit samples are incorporated into the system as deemed appropriate.
- Maintain a record of performance and system audits and inform the Project Manager of any problems encountered in the analytical procedures.
- The QA Officer in conjunction with the Project and Laboratory Managers will formulate recommendations on corrective action procedure to correct any deficiency in the analytical protocol, data, or sampling.

2.05 Assistant Project Managers

The management team for this project will draw upon the technical expertise and experience of a number of different individuals. The project team will consist of multidisciplined personnel with expertise in Aerial Photograph interpretation, hydrogeology, geophysical surveys, chemical characterization, soil science, wet chemistry and toxicology. The firms toxicologist will be responsible for the development of both the Safety Plan and the Risk Assessment.

2.06 Manager of Analytical Services

The Laboratory Manager is responsible for the overall administration of the analytical operations at O'Brien & Gere. The section

group leaders handle the day to day operations and scheduling and report to the manager.

The Laboratory Quality Assurance Manager, shown in Figure 4, is responsible for the implementation, monitoring and supervision of the QA/QC program. He assures that the program is conducted in strict adherence to procedures and requirements outlined for this program. He reports to the Laboratory Manager and interacts daily with other group leaders and laboratory staff. His duties include:

- Insuring laboratory custody procedures are followed.
- Monitors daily precision and accuracy records.
- Maintains copies of all procedures routinely used.
- Implements correction measures if results are "out of control"
- Reschedules analysis based upon unacceptable accuracy or precision data.

The Laboratory QA Manager will conduct an initial data validation and assessment on analytical results from the four laboratories performing the analysis. A final data validation and assessment will be conducted by USEPA's ~~QA Officer~~. The USEPA QAO will also provide PE samples where required and review the analytical results.

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SECTION 3 - QUALITY ASSURANCE OBJECTIVES

3.01 Overall Objectives

The general quality assurance objective for analyzed measurement data is to ensure that environmental monitoring data of known and acceptable quality are provided.

For this project, the specific objectives for measurement data in terms of precision, accuracy and compatibility are similar to the objectives established for the Statement of Work for the U.S. EPA Contract Laboratory Program (CLP), viz.: The purpose of the QA/QC program....is the definition of procedures for the evaluation and documentation of subsampling, analytical methodologies, and the reduction and reporting of data. The objectives are to provide a uniform basis for subsampling, sample handling, instrument condition, methods control, performance evaluation, and analytical data generation and reporting." Specific objectives for CLP and non-CLP analysis are included in Table 10. This QAPP for sampling, analysis and data handling is consistent with the requirement set forth by the U.S. Fish and Wildlife Service, as well as all State and Federal EPA requirements. Specific QA/QC is identified for those parameters requiring special analytical services.

3.02 Field QC Objectives and Procedures

Field functions such as; magnetometer and electromagnetic terrain conductivity services performed during Phase I are activities which do not include sample collection, but involve measurements where quality

assurance concerns are appropriate. The primary objective in activities such as these is to obtain reproductable measurements consistent with their intended use.

The objective of sampling procedures is to obtain samples that represent the environmental matrix being investigated. Trace levels of contaminants from external sources will be eliminated through the use of good sampling techniques and proper selection of sampling equipment.

A detailed description of sampling procedures is presented in the Site Sampling Plans for Phase I (December 1985) and Phase II (April 1986). Source material used in developing the sampling plan included the following:

Technical Support Documents

- Samplers and Sampling Procedures for Hazardous Waste Streams (EPA-600/2-80-180)
- Test Methods for Evaluating Solid Wastes (EPA SW 846-1980)
- User's Guide to the EPA Contract Laboratory Program
- EPA Technique Monographs
  - 15--Purposes and Objectives of Sampling
  - 16--Water Sampling Methods
  - 17--Soil and Sediment Sampling Methods
  - 18--Sampling of Biological Specimens
  - 19--Methods of Collecting Concentrated (Hazardous) Samples
  - 20--Container Opening Techniques
  - 22--Sample Handling, Packaging, and Shipping Procedures

The Site Sampling Plans include the following protocols and documentation.

- Number of locations to be sampled
- Sampling procedures to be used at the site
- Tests to be completed at each sampling location
- Sampling equipment required at the site
- Sample containers required at the site
- Preservation methods to be used at the site for various types of samples
- Reagents, etc., required at the site for sample preservation
- Shipping containers required at the site
- Chain-of-custody procedures to be used at the site
- Shipping methods and destinations, marking instructions, special labels, etc.

3.03 Field QC Audits

Blanks and duplicate samples will be collected as part of the QA/QC program. Blanks are employed to ensure that neither glassware nor procedural contamination has occurred. Additionally, they are utilized to evaluate ambient site conditions which may cause sample contamination. If positive interferences occur, the Quality Assurance Officer will recommend to the Project Manager that sample collecting and handling procedures be technically reviewed to eliminate such sample contamination.

Duplicate samples are treated throughout as two unique samples. The results of duplicate analyses provide information on the overall precision of both the sampling and analytical programs.

Field duplicate, spikes and blanks for Phase II are summarized in Table 6 of the Phase II Site Operations Plan (Oct. 1986). As shown in Table 6B, field QA/QC samples are scheduled by analysis sets and provide for adequate number of field duplicates and blanks based on sample types and analytical parameters. These scheduled duplicates and blanks will be closely followed, although modifications will be made as necessary in the field depending on actual sample collection and shipment of batches of samples. Laboratory matrix duplicates and matrix spikes are included in Table 10.

3.04 Accuracy, Sensitivity and Precision of Analysis

Samples collected during Phase II will be analyzed using procedures presented in Table 10. Additionally, Table 10 contains, method detection limits, audit, frequency and control limits for all Phase II parameters shown in Tables 2B and 7.

SECTION 4 - SAMPLING PROCEDURES

4.01 Objective

The objective of this Sites Sampling Plan (SSP) is to document the sampling locations, procedures and practices that will be used in the Remedial Investigation sampling program to be conducted at Crab Orchard National Wildlife.

It has been determined that the sampling and analysis program at Crab Orchard National Wildlife Refuge will be accomplished in two phases.

Phase 1 has been completed and is the basis for determining whether additional sampling is necessary. Phase II will be employed to define the extent of contamination (both vertically and laterally) of any site identified during Phase 1 as a area of concern. The information obtained during Phase II will be used in conjunction with Phase II results in evaluating the remedial options.

In general, the analytical effort associated with Phase II will be less than that of Phase I, because the results of the initial effort will assist in diminishing the total number of sites and reducing both organic and inorganic constituents of concern.

4.02 Types of Samples

Various matrices will be sampled and analyzed as part of the Remedial Investigation. These include the following:

1. Waters: including ground waters, surface streams, raw and finished water supplies, pond waters and waters from Crab Orchard Lake.
2. Sediments: from streams, ponds and Crab Orchard Lake.
3. Soils: including soils potentially affected by surface spillage and fill material from sites of past disposal activity.
4. Air: as part of the site safety program.
5. Biota: including fish, turtles and crayfish.

For the most part, all samples will be obtained as single grab samples. However, at some sites, areal soil composites will be prepared. Compositing procedures are discussed below. Phase II sampling locations are identified in the Site Operations Plan (October, 1986).

4.03 Compositing Procedures

Areal composites of water samples (along stretches of streams, surfaces of ponds or depth composites in Crab Orchard Lake) will be prepared by combining equal volumes of grab samples at each location. The nine water and sediment locations identified in the Site Operation Plan (October 1986), and one control site in the western end of Crab Orchard lake will be sampled during Phase II, to determine the extent of water and/or sediment contamination.

A composite water sample from each of the 10 Lake locations will be obtained as follows: discrete samples from the surface, mid-depth and approximately six inches from the bottom will be taken using a

w/ h what ?

stainless steel Kemmerer. The Kemmerer will be rinsed prior to each sample collection. Equal aliquots from each of the three depths at each site will be composited and preserved.

The question of thermocline most often applies to deep bodies of water. Crab Orchard lake has a mean depth of 3 meters and a maximum depth of 10 meters in the western portion of the lake. The eastern portion of the lake is shallow, hence, is not expected to have a significant change in temperature with depth and therefore the above compositing method will accomplish the Phase II objective, to define the extent of contamination both laterally and vertically.

Where applicable, areal composites of soil/sediment samples will be obtained by combining equal volumes of grab samples from predetermined locations. Soil/sediment grabs will be obtained using standard geological tools (shovels, scoops, etc.) to a depth of 3 inches. The discrete samples will be placed into a (disposable) aluminum pan and homogenized using a large stainless steel spoon. The composited sample is then placed into appropriate (pre-labeled) sample containers and refrigerated (0-4°C).

4.04 General Sampling Locations and Numbers

Sample Locations

Phase I sampling locations were determined in the field during a site reconnaissance visit on March 26-28, 1985. They are presented in the Site Sampling Plan (Dec. 1985). The Phase II

sampling locations are identified in the Site Operations Plan (October 1986). A log book listing the various samples to be collected will be prepared for use on-site. The log book will also contain the type of sample and analytical matrix for each of the samples to be collected. Pre-printed peel-off labels will be included in the log book for tagging the various containers to be used for sample collection. The sample team leader will be responsible for determining the exact sampling location and recording the location in the field sampling notebook. The location will be described in the log book with a sketch that includes distances from numbered field reconnaissance stakes and other landmarks. The rationale of selecting a sampling location will also be included. All sampling locations will be photographed.

Sample Numbering System

A sample numbering system will be used to identify each sample taken during the remedial investigation sampling program. This numbering system will provide a tracking procedure to allow retrieval of information regarding a particular sample and to assure that each sample is uniquely numbered. A listing of the sample identification numbers will be maintained by the sample team leader.

4.05 Sampling Equipment and Sampling Procedures

Soil Sampling

Equipment and supplies for the Phase II sampling activities are enumerated in Attachment 4.

All sampling equipment, except disposables, such as aluminum pans, will be decontaminated between sampling sites. General Decontamination procedures for sampling equipment are given on pages 9 through 11 of this section.

Soil samples will be collected from identified spots around the Refuge and during the installation of additional groundwater monitoring wells. Samples will be collected in general accordance with the split spoon sampling procedure (ASTM D1586-67), using 2-inch OD split spoon samplers.

Groundwater Studies and Sampling

Of the monitoring wells scheduled for the Crab Orchard NWR, nine shallow wells were installed at Site Nos. 15, 28, 29, 30 and 31 in Phase I. Eleven shallow and four deep monitoring wells will be installed at Site Nos. 17, 22, 28, 29, 32 and 33 during Phase II.

Two shallow wells were already installed by ~~Illinois~~ EPA prior to Phase I at Site #32. Three additional wells, including one deep well, will be installed at Site No. 32 in Phase II. At Site No. 28, one additional shallow and one deep well will be installed in Phase II. At Site No. 29 a deep well will be installed. Locations of all wells are identified in the Phase II Site Operations Plan, November 1986.

a) Well Installation

All monitoring wells to be installed in Phase II will be constructed of Type 316 stainless steel well screen and TIMCO

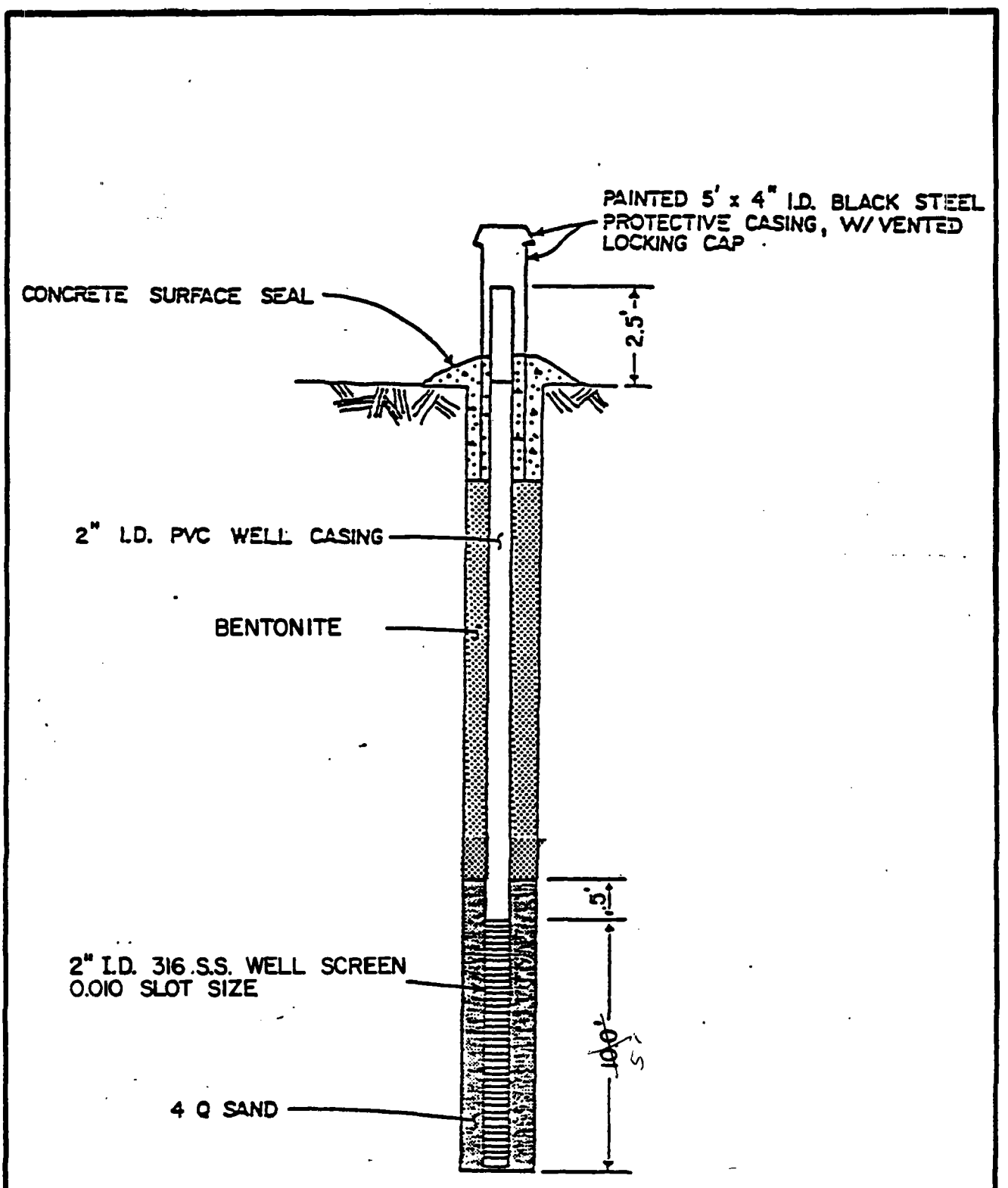
1. Re ID of how decide how deep to screen wells.
2. Split spoon sample continuously to bedrock.
3. Qualified person classify soil.
4. Analyze a number of samples for grain size and Atterberg limits.
5. Store all samples not analyzed.

Worded in accordance with 4.07.

or equivalent NSF approved P.V.C. riser casing that will extend from the screened interval to 2'-3' above existing grade. These well materials will be steam cleaned prior to installation. Other materials utilized for completion will be washed silica sand (Q-Rock Number 4 or approved equivalent) bentonite grout, Portland Cement and a protective steel locking well casing and cap with locks.

The monitoring well installation method for 2" wells installed within unconsolidated sediments shall be to place the screen and casing assembly into the hollow stem auger string once the screen interval has been selected. At that time a washed silica sand pack will be placed if required to prevent screen plugging. If a sand pack is not warranted, the auger string will be pulled back to allow the native aquifer material to collapse 2-3' above the top of the screen. Bentonite will then be added to the annulus between the casing and the insider auger wall via a tremle pipe to insure proper sealing. Bentonite will continue to be added during the extraction of the augers until the entire aquifer thickness has been sufficiently sealed off from horizontal and/or vertical flow above the screened interval. During placement of sand and bentonite frequent measurements will be made to check the height of the sand pack and thickness of bentonite-layers by a weighted drop tape measure.

A vented protective steel casing shall be located over the PVC standpipe extending two (2) feet below grade and



TYPICAL OVERBURDEN MONITOR WELL

O'BRIEN & GERE ENGINEERS, INC.

2-3' above grade secured by a Portland Cement seal. The casing will be cleaned and rinsed prior to use. The cement seal shall extend laterally at least one foot (1') in all directions from the protective casing and shall slope gently away to drain water away from the well. A vented steel cap will be fitted on the protective casing and a steel hasp shall be welded on one side of each steel casing so the cap may be secured with a steel lock.

All drilling equipment and associated tools will be decontaminated between completion of each well to prevent the transfer of contaminants between well locations via the drilling equipment. The decontamination will be accomplished using a high pressure steam cleaner. An area away from the well drilling operations shall be selected for completion of this task.

The supervising geologist shall specify the monitoring well design to the Drilling Contractor before installation.

b) Well Development

All wells will be developed or cleared of fine grained materials to ensure that the screen in transmitting ground water properly. The development will be completed using air surging or bailing methods until the well yields sediment-free water. If air surging methods are used, new polyethylene tubing will be used for each well. Similarly, new

polypropylene rope will be connected to a clean bailer for each well. The water removed from the wells will be allowed to discharge to the ground surface.

c) Water Level Measurements

The locations and elevations of all of the newly installed wells will be surveyed. This survey will be tied in to an existing survey of the Refuge if possible. All elevations will be determined based on USGS benchmarks. Ground water elevations have been measured on three occasions at all wells installed during Phase I. Additional water level measurements will be taken during Phase II from the existing wells and those that will be installed.

To ensure accuracy of the water level measurements on each occasion, the measurements shall be made from the same reference point located on the top of the riser casing each time. This reference point shall be marked and its elevation will be determined during the survey.

d) Ground Water Sampling

Ground water samples will be collected from each of the newly installed monitoring wells. A separate stainless steel bailer will be designated for each of the sites. This bailer will be thoroughly cleaned between use at each well within a site area. The decontamination procedure for the bailers will consist of a soapy water wash followed by a clean water

rinse. The bailer will then be rinsed with a dilute methyl alcohol solution followed by a final rinse with distilled water.

Prior to collection of the samples, each of the wells will be purged until a constant conductivity is maintained (generally 5 to 10 well volumes). The wells will then be allowed to recover if necessary and the sample will be collected using a clean stainless steel bailer. The collected samples will be placed into appropriate sample containers and will be preserved as necessary. An aliquot will be filtered in the field through a 0.45 micron filter prior to being preserved for dissolved metal analysis. Another portion will be preserved unfiltered for total metals analysis. All sample containers will be labeled and placed in coolers with ice for shipment to the laboratory. Appropriate chain-of-custody procedures will be followed throughout the transport of the samples.

e) Permeability Testing

In-situ permeability tests will be conducted on each of the newly installed monitoring wells to estimate the hydraulic conductivity of the material in which the well is installed. Prior to initiating this test the static water level in the well will be measured.

The in-situ permeability tests will be conducted by rapidly inserting a solid pieces of teflon or pvc into the water column in the well, thereby displacing the water column

upward and creating a potential for flow from the well to the surrounding aquifer. The rate of decline of the water level within the well is then monitored as it comes into equilibrium with the aquifer.

After the water level approaches the static water level, the rod will be removed. This will lower the water level in the well to a depth lower than the water table in the surrounding aquifer and thus create a potential for ground water flow into the well.

This recovery will also be monitored until the water level is close to the static level measured prior to conducting the permeability test.

Ground water levels during the tests will be monitored using an Enviro-Labs Data Logging System which employs a conventional analog signal generating pressure transducer that directly measures feet of hydraulic head to the one-hundredth (0.01) of a foot. The collected data will be analyzed using Hvorslev's method.

Waste Sampling

The Area 9 Landfill and possibly Water Tower Landfill are the only sites of the Refuge where waste materials are being sampled. All other sites represent sampling of matrices potentially affected by dispersed contaminants. There are special safety concerns posed by the sampling of waste materials at Area 9 because of the possible presence of explosives residues or even undetonated cartridges. Similar concerns exist at other sampling sites, but

sampling elsewhere is limited to within 1 foot from the surface. Soil borings at Area 9 will employ split spoon sampling procedures. Drilling personnel will be required to be removed at least 100 ft. from the drill rig during advancement of the augers. This is further discussed in the SHSP.

Field Blanks

Field Blanks for sediment and soil samples will consist of analytical grade diatomaceous earth. For water samples, ultrapure distilled/deionized water will be used. The field blank sample will be placed into the appropriate decontaminated sampling equipment, removed from the equipment, and then placed into sampling containers. Field blanks for the lake water columns using the Kemmerer will also be collected. Field blanks are identified in Table 6B.

Duplicate Samples

Duplicate samples are defined as two distinct samples taken from the same location at similar times using identical sampling equipment that has been decontaminated in a similar manner. However, duplicate samples of soil cores will consist of a given core homogenized, divided equally and submitted for analysis as two distinct samples. Duplicate samples are identified in Table 6B.

Split Samples

A number of samples will be split with a representative of the FWS for analysis. Split samples are defined as one distinct sample

that is divided equally and sent to two different laboratories for analysis. Soils will be field homogenized in a clean aluminum pan prior to splitting. Water sample splits will be duplicates.

4.06 General Decontamination Procedures

Decontamination of personal gear (boots, gloves, and waders), sample jars and sampling equipment will be as follows (see also attached materials to the SHSP):

1. Personal gear or sample containers will be washed in a bucket or tub filled between 50 and 75 percent with a trisodium phosphate (TSP) solution (2 lbs of TSP per 10 gallons of clean water). The entire exterior surface of the article undergoing decontamination will be brushed. If PCB's are expected to be present, 4 lbs of sodium bicarbonate per 10 gallons of water will be added to the washing solution.
2. Personal gear or sample containers will be rinsed in a bucket or tub filled between 50 and 75 percent with clean water. The entire exterior surface of the article undergoing decontamination will be completely brushed.
3. All wash and rinse water will be disposed of in a properly marked and sealed container. All such containers of wastewater will be stored in a secure area on-site and properly disposed of during the remedial action phase.

4.07 Sampling Equipment

1. Sampling equipment will be washed in a bucket or tub filled between 50 and 75 percent with a TSP solution (2 lbs of TSP per

10 gallons of clean water). The entire exterior surface of the article undergoing decontamination will be completely brushed. Interior wetted surfaces will be washed as required. If PCB's are expected to be present, 4 lbs of sodium bicarbonate will be added to the washing solution. Drilling equipment, augers and split spoon samplers will be decontaminated by steam cleaning using clean water.

2. Contaminated sampling equipment will be rinsed in a bucket or tub filled between 50 and 75 percent with methanol. The entire exterior surface of the article undergoing decontamination will be completely brushed. Interior wetted surfaces will be rinsed as required. If PCB's are present, the first rinse should be carried out with a hexane solution.

3. Following step 2 above, all sampling equipment will be rinsed in a bucket or tub filled between 50 and 75 percent with distilled water. The entire exterior surface of the article undergoing decontamination will be completely brushed. Interior wetted surfaces will be rinsed as required.

4. Collect all wash and rinse water in a properly marked and sealed container. Wash and rinse water will be analyzed relative to its hazardous waste characteristics and disposed of in accordance with all applicable state and federal regulations. Drilling soils and water as well as discarded protective clothing will be treated similarly.

4.08 Documentation

Site Location Procedure

Following sampling location identification, a wood stake (approximately 2" X 2" X 24") will be driven into the ground, allowing approximately 8 to 10 inches of the stake to remain visible above ground. The top portion of the stake will be painted orange and labeled for identification. The label will contain sample number and sample type. The location of each stake will be recorded. Sample locations will eventually be surveyed and tied into the site grid system.

4.09 Photographs

Photographs (35mm, color slides) will be taken to illustrate sampling locations. Photographs will show the surrounding area and reference objects which help to locate sampling sites. The picture number and roll number (if more than one roll of film is used) will be logged in the field notebook to identify which sampling site is depicted in the photograph. The film roll number will be identified by taking a photograph of an informational sign on the first frame of the roll. This sign would have the job and film roll number written on it to identify the pictures contained on the roll.

4.10 Field Notebooks

Field notebooks will provide the means of recording data on collecting activities performed at a site. As such, entries will be described in as much detail as possible so that anyone going to the site could reconstruct a particular situation without reliance on memory.

Field notebooks will be bound. Notebooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each notebook will be identified by the project-specific document number.

The cover of each notebook will contain:

Person or Organization to whom the book is assigned.

Book Number

Project Name

Start Date

End Date

Entries into the notebook will contain a variety of information. At the beginning of each entry, the date, start time, weather, all field personnel present, level of personal protection being used onsite, and the signature of the person making the entry will be entered. The names of visitors to the site, all field sampling team personnel and the purpose of their visit will be recorded in the field notebook.

All measurements made and samples collected will be recorded. All entries will be made in ink with no erasures allowed. If an incorrect entry is made, it will be crossed out with a single strike mark. Wherever a sample is collected or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The film roll number and number of photographs taken of the station will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the procedures documented in this plan. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. In addition, the cooler number into which the sample is placed in the field will be recorded. Sample numbers will be assigned prior to going onsite. Duplicates, which will receive an entirely separate sample number, will be noted under sample description. Significant field notebook entries (samples collected, significant observations) shall be countersigned by another member of the project team.

4.11 Control of Contaminated Sampling Materials

Disposable sampling and safety equipment and excess samples may be generated during sampling operations. These materials will be placed into drums (separate drums for solids, decontamination liquids, debris, and disposable equipment).

Decontamination liquids should also be separated based on those containing solvents (acetone, hexane, etc.) and those containing only detergents (TSP, etc.). The drums will be sealed, labelled and properly stored in a secure area for proper, legal disposal during the remedial action phase. Bailed well water and contaminated drilling spoils will be drummed for proper storage in a secure area.

Sample Control

Serialized sample tags will be used to label each sample for analysis. Chain-of-custody records will be completed for all samples according to EPA requirements and procedures set forth in NEIC Policies and Procedures EPA-330-19-78-001R (Revised 1986). Custody seals will be placed on all shipping coolers containing samples.

4.12 Sample Containers and Sample Preservation

Required sample containers, filling instructions and preservation procedures are listed below. The collected samples will be kept out of direct sunlight and, after decontamination and labeling, will be placed in coolers for shipment to the analytical laboratory.

Sample containers will be supplied by the O'Brien & Gere's laboratory. In order to insure both sufficient quantity and proper container cleanliness the contract laboratory will order these supplies from I Chem Research, Inc. located in Hayward California. When ordering the containers the contract laboratory will specify pre-cleaned jars with teflon liners. The types of containers are as follows:

Sample Shipping

Samples will be packed and labelled according to DOT regulations and protocols outlined in the Site Sampling Plan dated June 1985. Samples will be shipped via a 24 hour delivery service to the analytical laboratory so that the samples can be extracted within allowable time limits.

SAMPLE CONTAINERS AND PRESERVATIVES

PARAMETERS	WATER & WELL	SOIL & SEDIMENTS
1. CLP HSL Full Analysis	see below	see below
2. CLP HSL Volatiles	40 ml vial (2)	40 ml vial (2)
3. CLP HSL Base/Neut/Acids	1 Liter glass	1 Liter glass
4. Nitrosamines (CLP, soil)	--	-- ?
5. Nitrosamines (low, water)	1 Liter glass (amber)	--
6. CLP HSL Pesticide/PCB	1 qt. glass (teflon)	1 qt. glass (teflon)
7. PCB's General	--	1 pt glass (teflon)
8. PCB's Low Level (water)	1 qt. glass (teflon)	--
9. PCB's Semi-low (sediment)	--	1 pt glass (teflon)
10. Metals - CLP HSL	1 pt plastic/HNO3	1 pt glass
11. Metals - NIPDWR	"	
12. Sp. - Mercury	1 pt plastic/HNO3	1 pt glass
- Cadmium - Flame	--	"
- Furnace	1 pt plastic/HNO3	--
- Chromium - Flame	--	1 pt glass
- Furnace	1 pt plastic/HNO3	--
- Magnesium - Flame	"	1 pt glass
- Lead - Flame	--	"
- Furnace	1 pt plastic/HNO3	--
- Arsenic - Furnace	"	1 pt glass
- Copper - Flame	--	"
- Furnace	1 pt plastic/HNO3	--
13. EP Toxicity - Cr	--	1 pt glass
- Cd, Cr, Pb	--	"
14. Cyanide	1 pt plastic/NaOH	--
15. Indicators - pH	1 pt plastic	--
- NH3, NO3, F	"	--
16. Explosives by HPLC	1/2 pt glass	1/2 pt glass
17. Lipids	--	--
18. PCDD/PCDF	--	1 pt glass teflon
19. Total Phosphorus	1/2 pt glass	1/2 pt glass
21. Percent Solids (soil/sed)	--	"

Transfer of Custody and Shipment

1. Samples are accompanied by a field Chain-of-Custody Record, Figure 5. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in a mobile laboratory, or at the laboratory.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment (for example, one for each field laboratory, one for samples driven to the laboratory). Shipping containers will be padlocked or sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information are entered in the bottom of form.

3. Whenever samples are split with a source or government agency, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case

where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

4. All shipments will be accompanied by the field Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and a copy will be retained by the Project Coordinator.

5. If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a Government Bill of Lading will be used. Air freight shipments are sent collect. Freight bills, Post Office receipts, and Bills of Lading will be retained as part of the permanent documentation.

Evidence Files

All documents/raw data from the individual laboratories performing specific analysis will be transferred at the end of this RI/FS to the Refuge Manager for the Fish & Wildlife Service, Crab Orchard NWR for safekeeping for a period of 10 years.

Containers

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interference with constituents, cost and availability. There are also various procedures for cleaning and preparing bottles depending upon the analyses to be performed on the sample.

SECTION 5 - SAMPLE CUSTODY

5.01 General

Sample custody procedures for this project will be in strict conformance with the procedures detailed in NEIC Policies and Procedures (EPA-3309-78-001-R). These procedures were established to comply with EPA requirements for sample control. They are documented in Attachment 4 to this QAPP.

All samples collected for analysis will be taken by chemists, physical science technicians, or other qualified personnel designated by O'Brien & Gere with specific instructions from the Project Manager. The FWS will take duplicate samples at a ratio of 1:10 for QA/QC purposes. All samples for residue analysis will be placed in the custody of the analytical chemist responsible for the analysis. The sample information will be recorded on the same report sheets if analyzed immediately. Stored sample (including archive portions) will be catalogued and stored may be audited by the QA Officer. Subsequent to approval of the conceptual design (Task 15), these archived samples will be returned to CONWR for disposal consistent with the remedial action plan.

5.02 Chain of Custody Procedures

The consequences of an uncontrolled hazardous waste site investigation are difficult to predict. There is a possibility that several years after the RI/FS is complete there will be litigation. For that reason, it is imperative that an accurate record be maintained and documented of sample collection, transport, analysis and disposal.

Therefore, chain of custody procedures are instituted and followed throughout the study.

Chain of custody procedures include field custody, laboratory custody, and evidence files. Samples are physical evidence and should be handled according to procedural safeguards. The project coordinator must be prepared to produce documentation that traces the samples from the field to the laboratory and through the analysis. The National Enforcement Investigation Center (NEIC) of the U.S. EPA defines custody of evidence in the following ways:

- In actual physical possession
- In view after being in physical possession
- In a locked repository
- In a secure, restricted area

Chain of custody records begin in the field when sample collection has been completed. See Figure 5, Section 4 "Chain of Custody Form" for a typical arrangement of the paper samplers use to complete their field logs. On that form, they note meteorological data, equipment employed during collection, evacuation techniques and any calculations, physical characteristics of samples, date, time of day and location, any abnormalities during sampling.

The sampler completes the custody form, packages the samples including the custody form, and seals the package with evidence tape. Shipment may be made by commercial vendors, and their policy is to document the transfer of the package within their organization. Therefore, when the sample arrives at the laboratory, the sample custodian

signs the vendors air bill or bill of lading. The sample custodian's duties and responsibilities upon sample receipt are:

- Document receipt of samples.
- Inspect sample shipping containers for presence or absence of custody seals, locks, evidence tape, container integrity.
- Record condition of shipping and sample containers in logs.
- Sign appropriate forms or documents.
- Verify and record agreement or disagreement of information on sample documents. If there is discrepancy, record the problem and notify the project officer.
- Label sample with laboratory sample number.
- Place samples in storage, including secure storage.

The hand-to-hand custody of samples in the laboratory is maintained through preparation and analysis. The analyst is required to log samples into and from secure storage as the analysis proceeds. Samples are returned to secure storage at the close of business. Log sheets incorporate options for multiple entries, because several people handle the samples throughout the analytical scheme.

The laboratory records may also be used as evidence in enforcement proceedings, therefore care must be exercised to properly complete, date and sign items needed to generate data. Copies of the following items are stored:

- Documentation of the preparation and analysis of samples, including copies of the analyst's notebooks.

- Bench sheets, graphs, computer printouts, chromatographic outputs, mass spectral outputs.
- Copies of all QA/QC data.
- Instrument logs showing date, time and analyst.
- Analytical tracking forms which record date, time, and analyst for each step of sample preparation and analysis.

Upon completion of analysis, the project officer or his assignee should commence assimilating all the field and laboratory notes. It is they who generate the evidence file for the project. The package is arranged in chronological order for ease of review. When all the information is gathered, the package is inventoried, numbered and stored for future reference.

The sample custodian logs in the samples on a log-in form (Figure 6) and notes the appropriate information, including sample identification and condition of the samples. Any inconsistencies in paperwork or comments on the condition of the samples are duely noted on the form and filed with the case. The analyst performing the logs out and in the samples from secure storage as the analyses are completed (Figure 7). To further document the custody of each sample, the analyst will complete Figures 8, 9, 10 and 11, Sample Preparation and Extraction Log, Surrogate Standard Work Sheet, GC Logbook, and GC/MS Logbook, respectively. In all cases the chemist or technician signs and dates the appropriate forms when handling the samples.

During the analysis, these forms will be maintained in a secure file. Following the completion of a group of samples all appropriate

forms and data sheets will be gathered and stored in the files. If necessary, the files will be purged of all the appropriate records and transmitted to the Project Officer.

DATE: 8-22-86

Figure 6
Log In Form

SAMPLE CUSTODIAN SIGNATURE: Anne Ecklett

DOCUMENT CONTROL # 1042.150.100 - 1042.150.200 1042.150.300

CIRCLE THE APPROPRIATE RESPONSE

1. Custody Seal — present/absent; intact/not intact
2. Chain-of-Custody — present/absent
3. Sample Tags — present/absent
   Sample Tag Numbers — listed/not listed on chain-of-custody
4. SMO Forms — present/absent

CASE NUMBER —

AIRBILL NUMBER —

DATE RECEIVED	TIME RECEIVED	CHAIN-OF-CUSTODY RECORD NUMBER	SMO SAMPLE NUMBERS	CORRESPONDING: SAMPLE TAG NUMBERS	CORRESPONDING: ASSIGNED LAB NUMBERS	DOES INFORMATION ON CUSTODY RECORDS, TRAFFIC REPORTS, AND SAMPLE TAGS AGREE?	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.
8-22-86	2:00pm	—	—	Sample #1	D0060	yes.	Red Evidence tape not on samples when received.
				Sample #2	D0061		
				Sample #3	D0062		
				Sample #4	D0063		
				Sample #5	D0064		
				Sample #6	D0065		
				Sample #7	A2315		
				Sample #8	A2318		
				Sample #9	A2488		
				Sample #10	A2489		
				Sample #11	D0070		
				Sample #12	D0071		
				Sample #13	D0072 (Red tape)		
↓	↓	↓	↓	Sample #13	A2490 (Brown)	↓	

4961 CL 1/84

LABORATORY SAMPLE NUMBER	REMOVED BY	DATE AND TIME REMOVED	REASON	DATE AND TIME RETURNED
D0071	LMB	8/28/86 8:15AM	Prof.	
D0070	"	" "	"	
D0060	N. HOWE	9-6-86 1:20	Nickle Analysis and Pb	9-6-86 2:00
61	↓	↓	↓	↓
62				
63				
D0060	N. Howe	9-7-86 12:30	Thallium Analysis and Mn	9-7-86 1:00
61	↓	↓	↓	↓
62				
63				
D0060	N. Howe	9-7-86 1:40	Sb Analysis and Zinc	9-7-86 3:45
61	↓	↓	↓	↓
62				
63				
D0060	N. Howe	9-8-86 12:30	Ba Analysis	9-8-86 2:00
D0061	N. Howe	9-8-86 12:30	Ba Analysis	9-8-86 2:00

LABORATORY SAMPLE NUMBER	REMOVED BY	DATE AND TIME REMOVED	REASON	DATE AND TIME RETURNED
DC062	N. Howe	9-8-86 12:30	Ba Analysis and Ag	9-8-86 2:00
DC063	N. Howe	9-8-86 12:30	Ba Analysis	9-8-86 2:00
DC060	N. Howe	9-8-86 2:30	Be Analysis	9-8-86 3:00
61	↓	↓	↓	↓
62				
63				
DC060	N Howe	9-9-86 10:30	Se Analysis	9-9-86 10:45
DC061	N. Howe	9-9-86 10:30	Se Analysis	9-9-86 10:45
DC062	N. Howe	9-10-86 9:00	Se Analysis	9-10-86 9:30
DC063	N. Howe	9-10-86 9:00	Se Analysis	9-10-86 9:30
DC060	N. Howe	9-19-86 8:00	Cu, V, Fe, AL, CD, CR, Be, As	9-22-86 3:30
61	↓	↓	↓	↓
62				
63				

LABORATORY SAMPLE NUMBER	REMOVED BY	DATE AND TIME REMOVED	REASON	DATE AND TIME RETURNED
		8-26-86	Sample Prep	ALL SAMPLE
D0064	TAA	9-8-86 8:00AM	↓	USED FOR ANALYSIS
D0065	TAA	9-9-86 8:00AM		
A2315	RW	9-16-86		
A2318	RW.			

SAMPLE PREPARATION AND EXTRACTION LOGBOOK

ANALYSIS	METHOD	PROJECT # Client	Job	Phase	CLIENT NAME
AE ( )	624 ( )				
BN ( )	625 ( )				
AE/BN ( )	CLP ( )				
VOA ( )	*Dioxin 613* ( )				
Pesticide/PCB ( )	Dioxin IFB ( )				
Other	Other				

PROJECT # Client-Job-Phase			SAMPLE #	DATE Received	DATE Extracted	FINAL EXTRACT Volume	FINAL EXTRACT Solvent	SAMPLE SIZE	SS SPIKE SOLUTION

Notes: FB = Field Blank
MB = Method Blank
MS = Matrix Spike of Sample #_____
MSD = Matrix Spike Duplicate of Sample #_____
D = Duplicate of Sample #_____

_____
Extracted By

_____
Received For Analysis By

Comments: _____

Figure 8
Page 2 of 2

SAMPLE TRACKING FORM

CASE NO. 6457 PAGE NO. 1 OF ____

SAMPLE NUMBER	SAMPLE PREPARATION					EXTRACTION		CONCENTRATION		CLEAN UP			CONCENTRATION		GC/MS
	DATE & TIME	ANALYST	SAMPLE SIZE	SPIKE	COMMENTS	DATE/TIME	ANALYST	DATE/TIME	ANALYST	DATE/TIME	ANALYST	TYPE	DATE/TIME	ANALYST	DATE/TIME/ANALYST
A3809	10-7-86 1400	RW	20g	100 uL spike I		10-8-86 8:00	PT	10-8-86 13:00	PT	10-10-86 1100	PT	N	10-10-86 300	PT	
A3810															
A3811															
A3812				100 uL spike II											
A3813															
A3814															
A3815															
A3816															
A3817															
A3818															
A3819								SAMPLE LOST							
A3820															
A4002					Dup of A3812										
A4001					METHOD BLANK										

SURROGATES STANDARDS

1. BNAs CLP

Surrogates Stock Solution #_____ AE SS Conc._____ BN SS Conc._____

Surrogate	Volume SS Stock Used	Conc. ug/ml	Nonogram Spk Added
Phenol d-5			
2-Fluorophenol			
2,4,6-Tribromophenol			
Nitrobenzene d-5			
2-Fluorobiphenyl			
Terphenyl d-14			
Other(s):			

2. VOAs CLP

Surrogates Stock Solution #_____ Conc._____

Surrogate Standards	Volume SS Stock Used	Conc. ug/ml	Nanogram Spk Added
4-Bromofluorobenzene			
1,2-Dichloroethane d-4			
Toluene d-8			
Other(s)			

3. Other CLPs _____

Figure 10

DATE _____ INSTRUMENT _____ DETECTOR/MODE _____

COLUMN _____ DETECTOR TEMP _____ °C

_____

IP TEMP _____ °C TRANSFER LINE _____ °C FURNACE TEMP _____ °C

TEMP PROGRAM: Oven ____ °C TO ____ °C At ____ °C/min SOLVENT/FLOW _____ ml/min

INITIAL HOLD ____ min FINAL HOLD ____ min CARRIER FLOW _____ ml/min

GAS 1/FLOW _____ ml/min

GAS 2/FLOW _____ ml/min

COMMENTS: _____

_____

_____

BOT# INJ#	Project # Client	Job	Phase	Sample #	Sample Description	Inj. Volume	Conductivity Attenuation	Comments

CAGAS NO. _____ SIGNATURE(S) _____

_____

Figure 11

GC/MS INJECTION LOGBOOK

Date _____ Instrument _____ Column _____	Project # Client	Job	Phase	Client Name

Bot. # Inj. #	Project # Client	Job	Phase	Sample #	Inj. Vol.	Analysis	Data File Number	Date	Time	DF	IS

Notes: FB = Field Blank
MB = Method Blank
MS = Matrix Spike of Sample # _____
MSD = Matrix Spike Duplicate of Sample # _____
D = Duplicate ~~of~~ Sample # _____
IS = Internal Standard(s)
DF = Dilution Factor

Analyzed by _____

Signatures(s) _____

Comments _____

SECTION 6 - EQUIPMENT CALIBRATION

6.01 Calibration Procedures

Equipment Calibration, References and Frequency

All field equipment used during this project will be calibrated and operated in accordance with manufacturer's instructions. Any field equipment used during this project that is not covered by the investigator's standard operating procedures will have a specific calibration and operation instruction sheet prepared for it.

A. General

Standards may be generally grouped into two classifications: primary and secondary. Primary standards include USP and NE drugs, NBS and ASTM materials, and certain designated EPA reference materials. All other standards are to be considered secondary.

B. Testing

1. Primary: No testing is necessary. Do not use if there is any physical indication of contamination or decomposition (i.e. partially discolored, etc.).
2. Secondary: Examine when first received either by comparison to an existing primary, or comparing known physical properties to literature values. The less stable standards will be rechecked at appropriate intervals, usually six months to one year.

C. Records

1. A records book will be maintained for each grouping of standards (i.e. pesticides, metals, etc.)
2. The record kept for each standard will include:
   a. Name and date received
   b. Source
   c. Code or lot number
   d. Purity
   e. Testing data including all raw work and calculations
   f. Special storage requirements
   g. Storage location
3. These records will be checked periodically as part of the Laboratory Controls Review.

Equipment

A. General

1. Each major piece of analytical laboratory instrumentation used on this project is documented and on file with the analytical laboratory.
2. A form is prepared for each new purchase and old forms will be discarded when the instrument is replaced.

B. Testing

1. Each form details both preventative maintenance activities and the required QA testing and monitoring.
2. In the event the instrument does not perform within the limits specified on the monitoring form, the Laboratory

Manager will be notified and a decision made as to what action to take.

3. If repair is deemed necessary, an "out of order" sign will be placed in the instrument until repairs are effected.

6.02 Calibration Records

A bound notebook will be kept with each instrument, requiring calibration, to record all activities associated with a maintained, QA monitoring and repairs program. Additionally, these records will be checked during periodic equipment review.

SECTION 7 - ANALYTICAL PROCEDURES

7.01 Laboratory Analytical Procedures

The analysis and methods detection limits for Phase II analytical parameters are given in Table 10 of this section. Specific procedures associated with parameters requiring special analytical services are provided as attachments.

When analyzing samples by the above standardized methods, the accuracy or precision of the data generated by the laboratory is determined through analysis of replicates, spiked samples, synthetic reference standard samples, and/or field or laboratory blanks along with each set of samples. Any interference are identified and documented.

In general, the methods accuracy is determined by spiking the sample matrix with the analyte at a minimum of three concentration levels. The range of the spiking levels is selected to bracket the concentration of interest. Percent recoveries of the spikes are calculated and are compared with synthetic standards. The methods precision is determined by analyzing a minimum of three replicates at each spiking level. The precision is evaluated by calculating the standard deviation.

The data generated is, whenever possible, input the laboratory base data management system. Analyst's work sheets are filed for one year as a temporary record. When approved and signed, data reports and pertinent information are reported to the client.

The analytical protocols for explosives in soils are presented in Attachment 5. Samples to be analyzed for chlorinated dioxins and dibenzofurans will be analyzed according to the procedure of Smith et al. (1984) or equivalent as presented in Attachment 6.

7.02 Field Procedures

Field analyses of surface and groundwater will consist of pH, specific conductance and temperature measurements. Samples collected during the Phase II will be shipped, following chain-of-custody procedures to O'Brien & Gere's laboratory for analyses.

SECTION 8 - DATA REDUCTION, VALIDATION, AND REPORTING

8.01 General

Laboratory facilities performing analysis on Phase II samples are identified in Table 8A (Section 2). Data reduction and validation will be incorporated into the in-house effort for all parameters.

8.02 Data Reduction and Reporting

The following data handling procedures are employed at O'Brien & Gere:

A. Data Production - A Hewlett-Packard Model 5995 and 5993 are used for the positive identification and quantification of sample extracts. Output from the determination is a total ion chromatogram recorded on thermal printer hard copy and cassette tape.

B. Data Reduction - Output from the GC/MS unit is digitized, stored in memory on cassette tape and processed for presentation in three formats:

1) A real-time total multiple ion mass chromatogram.

2) A post-run integration report contains the following:

   a. Retention time

   b. Response factor

   c. Primary, secondary, and tertiary ion with their corresponding abundance

   d. Quantitation ion

e. Reference library name

f. Concentration

3) A visual comparison of the subject mass spectral output to the library compound.

C. Data Transcription - The post integration report contains the following:

1) Listing of all compounds.

2) Relative retention times.

3) Relative response factor to their internal standards.

4) Concentration of compounds, surrogate and internals.

Quality Control/Quality Assurance data such as resolution and calibration standards and DFTPP spectra are also processed and stored in the above manner.

D. Data Verification - The processed transcribed information and the hard copied raw data are now evaluated by the Group Leader to verify the validity of the data and determine whether reinjection or additional cleanup steps are required. The results of the evaluation are recorded in a notebook and inputted into the Sample Status File.

E. Distribution - Following final review the GC/MS Group Leader and Manager of Analytical Services, the results of the analytical determination are shipped to the Contractor. The format used for presentation of data are the presented in the IFB forms. Additional data such as copies of raw data and chromatograms are provided upon request.

F. In-House Storage - Results of all analytical determinations are stored in the RTE6 computer. Raw data tapes are logged into the computer on a separate file and listed by tape number and its contents. The data tapes are stored indefinitely. Should a request be made for a particular raw data tape, the tape is copied and the copy is kept in the archive while the original is sent to the Contractor. All notebooks are also archived and stored in the O'Brien & Gere Central File.

Reporting

Once a sample has been tagged and input into the laboratory data management system, we have the ability to determine its exact status. With the available maintenance programs, and tracking forms, the group leaders can trace the progress of one sample or an entire group of samples. Therefore, a client is able to receive partial data before the entire program is complete.

For a program that covers the course of several months or years, it is imperative that interim reports be submitted. It is anticipated that turnaround for a batch of samples will be 40 days from sample arrival. The RTE6 computer system, with the Aquarius software will generate a final report following injection and data evaluation. Therefore, if specific sample information is required prior to submission of the case, we would be able to satisfy EPA's needs.

Of course there may be certain instances where faster turn-around would be dictated and we shall make every attempt to meet

those needs. Our past experience on programs of this size have proven our capability to supply information in a timely manner.

8.03 Data Validation

Prior to submittal of the data to the Project Manager for his review, data will be validated by the individual laboratory group leaders and/or Manager.

The validation process by group leaders will include the review of spike recoveries, surrogate recovery, comparability of duplicate analysis and field blank integrity. Additionally, the group leaders will check for the adherence to accuracy and precision criteria, unusually high or low parameter values and possible transmittal errors.

The Laboratory Quality Assurance Manager identified in Figure 4 (Section 2) will perform validation of the data from all laboratories.

The requirements to be checked in validation, in order, are as follows:

I. Sample Holding Times

II. Calibration

   a. Initial Calibration and Calibration Verification

   b. Continuing Calibration Verification

   c. Calibration Blank

III. Blanks

   a. Laboratory preparation blank

b. Field blank

c. Procedural Blank

IV. Interference Check Sample Analysis

V. Laboratory Control Sample Analysis

VI. Specific Sample Results

a. Duplicate Sample Analysis

b. Spiked Sample Analysis

c. AA/QC Analysis

1. Duplicate Injections

2. Analytical Spikes

d. ICP QC Analysis

e. Sample Result Verification

VII. Field and Other QC

VIII. Quarterly Submissions

IX. Overall Case (Batch) Assessment

The reviewer will compare what was actually performed by the laboratory to the requirements of the protocols and program objectives. The intent is to review all the deliverables for completeness and all the raw data anomalies consistent with methods used by USEPA CRL.

SECTION 9 - INTERNAL QUALITY CONTROL PROCEDURES

9.01 Quality Control/Quality Assurance Objectives

The quality control objectives for the project are listed with each matrix in Table 10. The requirements for each group of compounds is different, therefore the listing identify the frequency and control limits for acceptability. Upon completion of analysis the results of quality control data will be reviewed to verify compliance with the criteria listed. The goal is to achieve compliance with the criteria, 88% completeness on this matrix spike and matrix spike duplicate. When results are reported to the project team, quality control data will be included in the package for everyones review. This will include the analysis of EPA standard reference materials where available to verify initial calibration of non CLP analysis. The criteria for acceptance will be ±10% of known values. Matrix spikes will monitor the methodology and discoveries will be compared to Exhibit E of the WA-85-177 CLP protocols. Matrix spike duplicates will be incorporated to be an indicator of the precision of sample results. The relative percent difference calculations will be compared to Exhibit E of the CLP protocol.

9.02 Field Sampling Quality Control

Field sampling crews will always be under the direct supervision of a crew chief with a minimum of a Bachelor's degree and five years sampling experience. New employees will be assigned to an experienced staff member and work under his/her direction.

Bound log books and appropriate data sheets will be used to document the collection of samples so that any individual sample can be traced back to its point of origin; sampler and sampling equipment.

Duplicate and blank samples (see Table 6B) will be collected at the same time, employing the same procedures, equipment and containers as the scheduled sample.

Additionally, duplicate samples will be packaged and shipped to the laboratory in the same manner as the required sample.

As specified in Section 8 of this QAPP the QAM will periodically review the results of the duplicate analyses and advise the Project Manager of any problems.

9.03 Field Analytical Procedures Quality Control

Field measurements of pH, temperature and specific conductance will be taken on water samples only. The pH meter will be checked against two known standard pH buffers (7 and 10) before and after each days use.

Temperature measurements will be made with a mercury-filled celsius thermometer. As a minimum, the thermometer will have a scale marked for every 0.1C, with marking etched on the capillary glass. Field operations will require a thermometer with a protective case to prevent breakage. The thermometer will be checked against a precision thermometer certified by the National Bureau of Standards (NBS) periodically.

Conductivity reading will be made with a portable specific conductivity meter. The meter will be calibrated against a 0.010 normal potassium chloride solution (KCL) at least once per day.

SECTION 10 - AUDIT PROCEDURES

The O'Brien & Gere Project Manager and the DOI Project Manager (Figure 3) will monitor the performance of the QA audit listed in this plan. O'Brien & Gere will conduct an initial audit of all analytical data, with the final audit performed by O'Brien & Gere and USEPA QAO.

The Quality Assurance Officer from USEPA, Region V will conduct a systems audit of the laboratories for Phase II analytical parameters. Procedures for the audits will be established by the QAO prior to such audit. Performance evaluation samples will also be provided by USEPA to appropriate laboratories.

O'Brien & Gere has designated a QA Officer as indicated in Figure 3 (Section 2.02). A performance audit, consisting of analysis of appropriate blanks, fortified samples and standard solutions will be performed prior to initiation of Phase II. O'Brien & Gere's QA Officer will maintain a record of such audits and will inform the FWS of significant deviations from established control limits. These audits will test not only the total system's response, but inherently all major measurement methods.

O'Brien & Gere's QA Officer will report to the Project Manager (Figure 3) and the FWS the result of assessment of: the accuracy, precision and completeness of the data, results of the performance and system audits, and any problems encountered in the analytical procedures. The QA Officer, in conjunction with the analyst, analyst's supervisor, and Project Manager will formulate recommendations to correct any deficiency in the analytical protocol or data. These

corrective measures will be in accord with ongoing good laboratory practices and the overall Quality Assurance Program.

SECTION 11 - PREVENTIVE MAINTENANCE

Preventive maintenance procedures will be carried out on all field equipment in accordance with the procedures outlined by the manufacturer's equipment manuals. Any field equipment used during this project that is not covered by the standard operating procedures will have a specific maintenance instruction sheet prepared for it.

SECTION 12 - DATA ASSESSMENT PROCEDURES

The O'Brien & Gere Laboratories QA/QC Manager will be responsible for data assessment. His assessment will be based upon instrument tuning criteria, surrogate recoveries, matrix spikes, matrix spike duplicates, duplicate analysis and reagent and field blank integrity. Procedures for data assessment will be consistent with those used by USEPA CRL.

The QA/QC Manager, with individual laboratory group leaders, will identify any data that should be rated as "unacceptable" or "preliminary", and take corrective actions, if deemed necessary.

Tentatively identified compounds (TIC's) will be brought to the attention of the Laboratory Manager who has the responsibility of deciding whether to require additional verification or discard the data.

The Laboratory QA/QC Manager has the responsibility of also assessing the quality of the data generated by outside contract laboratories. The Laboratory QAM will review both the analytical data and QA/QC reports from external laboratories and will report any inconsistencies to the Project QAO along with recommendations concerning the acceptability of the data.

Finally, all analytical data will be submitted to and assessed by the USEPA, Region V and FWS in accordance with their standard procedures.

SECTION 13 - CORRECTIVE ACTION PROCEDURES

Corrective action procedures that might be implemented from audit results or upon detection of data unacceptability are developed on a case-by-case basis.

The actions may include:

- Reanalyzing samples if holding time requirements have not been exceeded.
- Altering field or handling procedures.
- Resampling.
- Using a different batch of sample containers.
- Recommending an audit of laboratory procedures.
- Accepting data with knowledged level of uncertainty.
- Discard data.

The O'Brien & Gere Project Manager will discuss corrective actions with the Regional Resource Contaminants Assessment Coordinator prior to initiating them.

SECTION 14 - QUALITY ASSURANCE REPORTS

Discussions of quality assurance problems and corrective actions taken will be included in the project monthly progress reports. The final RI report and the final FS report will contain separate QA sections that summarize data quality information collected during the project. Specifically, the reports will include:

1. USEPA QAO system audit.
2. QAO report to FWS on accuracy, precision, completeness of data and results of performance and system audit.
3. Report to FWS and USEPA on results of data assessments.

Tables

O'BRIEN & GERE

TABLE 1

CRAB ORCHARD REFUGE

STUDY SITES
(Phases I and II)

Site #	Type	Name
Group 1		
3	Surface Soil	Area 11 South
4	Surface Soil	Area 11 North
5	Pond	Area 11 Acid Pond
Group 2		
7	Surface Water	D Area SE Drainage
7A	Surface Soil	D Area North Lawn
8	Surface Water	D Area SW Drainage
9	Surface Water	P Area NW Drainage
10	Surface Water	Waterworks North Drainage
11	Surface Water	P Area SE Drainage
11A	Surface Soil	P Area North
20	Surface Water	D Area South
Group 3		
12	Surface Soil	Area 14 Impoundment
13	Surface Soil	Area 14 Change House Site
14	Surface Water	Area 14 Solvent Storage
Group 4		
15	Pond	Area 7 Plating Pond
16	Surface Soil	Area 7 Industrial Site
Group 5		
17	Landfill	Job Corps Landfill
Group 6		
18	Surface Soil	Area 13 Loading Platform
19	Surface Soil	Area 13 Bunker 1-3
30	Control	Munition Control Site
Group 7		
21	Landfill	Southeast Corner Field
Group 8		
22	Surface Water	Old Refuge Shop
24	Surface Water	Pepsi-West
25	Surface Water	COC at Marion Landfill
26	Surface Water	COC below Marion STP
27	Surface Water	COC below 157 Dredge Area

TABLE 1
(Continued)

CRAB ORCHARD REFUGE

STUDY SITES

Site #	Type	Name
Group 9		
28	Landfill	Water Tower Landfill
Group 10		
29	Landfill	Fire Station Landfill
Group 11		
32	Landfill	Area 9 Landfill
33	Surface Soil	Area 9 Building Complex
Group 12		
34	Lake	Crab Orchard Lake
Group 13		
31	Control	Refuge Control Site

NOTES:

1. The names of sites 3, 4, and 12 have been changed from their previous descriptions as landfills to reflect the absence of any buried materials.

2. Sites 30 and 31 are included only as control sites.

TABLE 2A

RI/FS ANALYTICAL PARAMETERS
(for Phase I, completed November 1985)

1. Purgeable Priority Pollutants
   (Screening and Full Analysis)

2. Acid Extractable Priority Pollutants
   (Screening and Full Analysis)

3. Base/Neutral Extractable Priority Pollutants
   (Screening and Full Analysis)

4. Pesticide/PCB Priority Pollutants
   (Screening and Full Analysis)

5. PCB's

6. Metals
   - ICP scan
   - Priority Pollutant Metals by AA Spec
   - Mercury

7. EP Toxicity

8. Cyanide 40

9. Indicators
   - pH (field)
   - Specific Conductance (field)
   - Total Organic Carbon
   - Total Organic Halogens

10. Explosives Residues by HPLC

11. Nitrogen Series: TKN, NH3N, NO3N

12. PCDD/PCDF
    (Screening and Full Analysis)

13. Cation Exchange Capacity

14. Total Phosphorus

15. Primary and Secondary Drinking Water Standards

16. Percent Solids (for soil/sediments)

Note: See Table 2C for list of parameters included within each Phase I parameter.

TABLE 2B

RI/FS ANALYTICAL PARAMETERS
PHASE II
(to be initiated)

1. CLP HSL Full Analysis
2. CLP HSL Volatiles
3. CLP HSL Base/Neut/Acids
4. Nitrosamines (CLP, soil)
5. Nitrosamines (low level)
6. CLP HSL Pesticide/PCB
7. PCB's General
8. PCB's Low Level (water)
9. PCB's Semi-Low (sediment)
10. Metals - CLP HSL
11. Metals - NIPDWR
12. Special
    - Mercury
    - Cadmium
    - Chromium
    - Magnesium
    - Lead
    - Arsenic
    - Copper
13. EP Toxicity
    - Cr
    - Cd, Cr, Pb
14. Cyanide
15. Indicators
    - pH
    - NH_3, NO_3, F
16. Explosives by HPLC
17. Lipids
18. PCDD/PCDF
19. Total Phosphorus
20. Grain Size
21. Percent Solids (soil/sed)

Note:

1. See Table 2D for the list of compounds in each parameter.
2. See Table 7 for site specific details on analysis.
3. See Table 10 for analytical procedures.

LIST OF CHEMICAL COMPOUNDS FOR PARAMETERS IN TABLE 2A
(for PHASE I, completed November 1985)

PURGEABLE PRIORITY POLLUTANTS

1 Chloromethane
2 Bromomethane
3 Dichlorodifluoromethane
4 Vinyl chloride
5 Chloroethane
6 Methylene Chloride
7 Trichlorofluoromethane
8 1,1-Dichloroethene
9 1,1-Dichloroethane
10 t-1,1-Dichloroethene
11 Chloroform
12 1,1,1-Trichloroethane
13 Carbon tetrachloride
14 Bromodichloromethane
15 1,2-Dichloropropane
16 t-1,3-Dichloropropene
17 Trichloroethene
18 Benzene
19 Dibromochloromethane
20 1,1,2-Trichloroethane
21 c-1,3-Dichloropropene
22 2-Chloroethylvinyl ether
23 Bromoform
24 1,1,2,2-Tetrachloroethane
25 Tetrachloroethene
26 Toluene
27 Chlorobenzene
28 Ethylbenzene
29 1,1 Dichloroethylene
30 t-1,2-Dichloroethylene
31 Bromochloromethane
32 Trichloroethylene
33 2-Bromo-1-chloropropane
34 Tetrachloroethylene
35 Acetone
36 Carbon disulfide
37 2-Butanone
38 Vinyl acetate
39 2-Hexanone
40 4-Methyl-2-pentanone
41 Styrene
42 Total xylenes

ACID EXTRACTABLE PRIORITY POLLUTANTS

1 Phenol
2 d6-Phenol
3 2-Fluorophenol
4 2,4-Dimethylphenol
5 2-Chlorophenol
6 2-Nitrophenol
7 4-Nitrophenol
8 4-Chloro-3-methylphenol
9 2,4-Dichlorophenol
10 2,4-Dinitrophenol
11 Pentafluorophenol
12 2,4,6-Trichlorophenol
13 2-Methyl-4,6-dinitrophenol
14 Pentachlorophenol

BASE/NEUTRAL PRIORITY POLLUTANTS

1 1,3-Dichlorobenzene
2 1,4-Dichlorobenzene
3 1,2-Dichlorobenzene
4 Hexachloroethane
5 Bis (2-chloroethyl) ether
6 Bis (2-chloroisopropyl) ether
7 N-Nitrosodi-n-propylamine
8 Nitrobenzene
9 Hexachlorobutadiene
10 1,2,4-Trichlorobenzene
11 Isophorone
12 Naphthalene
13 Bis (2-chloroethoxy) methane
14 Hexachlorocyclopentadiene
15 2-Chloronaphthalene
16 Acenaphthalene
17 Acenaphthene
18 Dimethyl phthalate
19 2,6-Dinitrotoluene
20 Fluorene
21 4-Chlorophenyl phenyl ether
22 2,4-Dinitrotoluene
23 1,2-Diphenylhydrazine
24 Diethylphthalate
25 N-nitrosodiphenylamine
26 Hexachlorobenzene
27 4-Bromophenyl phenyl ether
28 Phenanthrene
29 Anthracene
30 Di-n-butyl phthalate
31 Fluoranthene
32 Pyrene
33 Benzidine
34 Butyl benzyl phthalate
35 Bis (2-ethylhexyl) phthalate
36 Chrysene
37 Benzo(a)anthracene
38 3,3-Dichlorobenzidine
39 Di-n-octylphthalate
40 Benzo(b)fluoranthene
41 Benzo(k)fluoranthene
42 Benzo(a)pyrene
43 Indeno(1,2,3-cd)pyrene
44 Dibenzo(a,h)anthracene
45 Benzo(g,h,i)perylene
46 N-Nitrosodimethyl Amine

PESTICIDES/PCB PRIORITY POLLUTANTS

1 Alpha-BHC
2 Gamma-BHC (Lindane)
3 Beta-BHC
4 Delta-BHC
5 Heptachlor
6 Aldrin
7 Heptachlor epoxide
8 Endosulfan I
9 4,4'-DDE
10 Dieldrin
11 Endrin
12 4,4'-DDD
13 Endosulfan II
14 4,4'-DDT
15 Endosulfan Sulfate
16 Endrin Aldehyde
17 Methoxychlor
18 Chlordane
19 Toxaphene
20 Arochlor-1016
21 Arochlor-1242
22 Arochlor-1221
23 Arochlor-1232
24 Arochlor-1248
25 Arochlor-1254
26 Arochlor-1260
27 Endrin ketone

LIST OF CHEMICAL COMPOUNDS FOR PARAMETERS IN TABLE 2A
(for PHASE I, completed November 1985)

PCDDs/PCDFs

1 Tetra-CDD
2 Penta-CDD
3 Hexa-CDD
4 Hepta-CDD
5 Octa-CDD
6 Tetra-CDF
7 Penta-CDF
8 Hexa-CDF
9 Hepta-CDF
10 Octa-CDF

EXPLOSIVES RESIDUES BY HPLC

1 HMX
2 RDX
3 1,3,5 TNB
4 1,3 DNB
5 NB
6 TETRYL
7 2,4,6 TNT
8 2,6 DNT
9 2,4 DNT

METALS (ICPs AND PP ATOMIC ABS.)

1 Aluminum
2 Antimony
3 Arsenic
4 Barium
5 Cadmium
6 Calcium
7 Chromium
8 Cobalt
9 Copper
10 Iron
11 Lead
12 Magnesium
13 Manganese
14 Molybdenum
15 Mercury
16 Nickel
17 Potassium
18 Selenium
20 Silver
21 Sodium
22 Tin
23 Titanium
24 Vanadium
25 Zinc

OTHERS

INDIATORS
1 pH
2 Specific Conductivity
3 Total Organic Carbon
4 Total Organic Halides

NITROGEN SERIES
1 Ammonia Nitrogen
2 Nitrate Nitrogen
3 Nitrite Nitrogen
4 Total Kjeldahl Nitrogen

CYANIDE

CATION EXCHANGE CAPACITY

TOTAL PHOSPHORUS

SAFE DRINKING WATER ACT STANDARDS

Primary Inorganic Chemicals
1 Arsenic
2 Barium
3 Cadmium
4 Chromium
5 Fluoride
6 Lead
7 Mercury
8 Nitrate
9 Silver

Organic Chemicals
1 Endrin
2 Lindane
3 Methoxychlor
4 Toxaphene
5 2,4-D
6 2,4,5-TP Silvex

Secondary Inorganic Chemicals
1 Chloride
2 Copper
3 Iron
4 Manganese
5 Sodium
6 Sulfate
7 Zinc
8 Corrosivity

LIST OF CHEMICAL COMPOUNDS FOR PARAMETERS IN TABLE 3
(for PHASE II, scheduled November 1986)

CLP HSL VOLATILES

1 Chloromethane
2 Bromomethane
3 t-1,2-Dichloroethene
4 Vinyl chloride
5 Chloroethane
6 Methylene Chloride
7 Styrene
8 1,1-Dichloroethene
9 1,1-Dichloroethane
10 Chloroform
11 1,1,1-Trichloroethane
12 Carbon tetrachloride
13 Bromodichloromethane
14 1,2-Dichloropropane
15 t-1,3-Dichloropropene
16 Trichloroethene
17 Benzene
18 Dibromochloromethane
19 1,1,2-Trichloroethane
20 c-1,3-Dichloropropene
21 2-Chloroethylvinyl ether
22 Bromoform
23 1,1,2,2-Tetrachloroethane
24 Tetrachloroethene
25 Toluene
26 Chlorobenzene
27 Ethylbenzene
28 Carbon Disulfide
29 1,2-Dichloroethane
30 Acetone
31 2-Butanone
32 Vinyl acetate
33 2-Hexanone
34 4-Methyl-2-pentanone
35 Total xylenes
~~36 Total xylenes~~

HSL CLP BASE/NEUTRAL/ACID EXTRACTABLES (SEMI-VOLATILES)

1 Phenol
2 2-Methylphenol
3 2,4-Dimethylphenol
4 2-Chlorophenol
5 2-Nitrophenol
6 4-Chloro-3-methylphenol
7 2,4-Dichlorophenol
8 2,4-Dinitrophenol
9 2,4,5-Trichlorophenol
10 2,4,6-Trichlorophenol
11 2-Methyl-4,6-dinitrophenol
12 Pentachlorophenol
13 4-Methylphenol
14 1,3-Dichlorobenzene
15 1,4-Dichlorobenzene
16 1,2-Dichlorobenzene
17 Hexachloroethane
18 Bis (2-chloroethyl) ether
19 Bis (2-chloroisopropyl) ether
20 N-Nitrosodi-n-propylamine
21 Nitrobenzene
22 Hexachlorobutadiene
23 1,2,4-Trichlorobenzene
24 Isophorone
25 Naphthalene
26 Bis (2-chloroethoxy) methane
27 Hexachlorocyclopentadiene
28 2-Chloronaphthalene
29 Acenaphthalene
30 Acenaphthene
31 Dimethyl phthalate
32 2,6-Dinitrotoluene
33 Fluorene
34 4-Chlorophenyl phenyl ether
35 2,4-Dinitrotoluene
36 2-Methylnaphtalene
37 Diethylphthalate
38 N-nitrosodiphenylamine
39 Hexachlorobenzene
40 4-Bromophenyl phenyl ether
41 Phenanthrene
42 Anthracene
43 Di-n-butyl phthalate
44 Fluoranthene
45 Pyrene
46 Butyl benzyl phthalate
47 Bis (2-ethylhexyl) phthalate
48 Chrysene
49 Benzo(a)anthracene
50 3,3-Dichlorobenzidine
51 Di-n-octylphthalate
52 Benzo(b)fluoranthene
53 Benzo(k)fluoranthene
54 Benzo(a)pyrene
55 Indeno(1,2,3-cd)pyrene
56 Dibenzo(a,h)anthracene
57 Benzo(g,h,i)perylene
58 2-Nitroaniline
59 3-Nitroaniline
60 4-Nitroaniline
61 4-Chloroaniline
62 Benzyl Alcohol
63 Benzoic Acid
64 Dibenzofuran

4 nitrophenol

CLP HSL PESTICIDES/PCB

1 Alpha-BHC
2 Gamma-BHC (Lindane)
3 Beta-BHC
4 Delta-BHC
5 Heptachlor
6 Aldrin
7 Heptachlor epoxide
8 Endosulfan I
9 4,4'-DDE
10 Dieldrin
11 Endrin
12 4,4'-DDD
13 Endosulfan II
14 4,4'-DDT
15 Endosulfan Sulfate
16 Methoxychlor
17 Chlordane
18 Toxaphene
19 Arochlor-1016
20 Arochlor-1242
21 Arochlor-1221
22 Arochlor-1232
23 Arochlor-1248
24 Arochlor-1254
25 Arochlor-1260
26 Endrin ketone

LIST OF CHEMICAL COMPOUNDS FOR PARAMETERS IN TABLE 3
(for PHASE II, scheduled November 1986)

PCDDs/PCDFs

1 Tetra-CDD
2 Penta-CDD
3 Hexa-CDD
4 Hepta-CDD
5 Octa-CDD
6 Tetra-CDF
7 Penta-CDF
8 Hexa-CDF
9 Hepta-CDF
10 Octa-CDF

EXPLOSIVES RESIDUES BY HPLC

1 HMX
2 RDX
3 1,3,5 TNB
4 1,3 DNB
5 NB
6 TETRYL
7 2,4,6 TNT
8 2,6 DNT
9 2,4 DNT

CLP HSL METALS

1 Aluminum
2 Antimony
3 Arsenic
4 Barium
5 Beryllium
6 Cadmium
7 Calcium
8 Chromium
9 Cobalt
10 Copper
11 Iron
12 Lead
13 Magnesium
14 Manganese
15 Mercury
16 Nickel
17 Potassium
18 Selenium
19 Silver
20 Sodium
21 Thallium
22 Vanadium
23 Zinc

NIPDWR METALS (40CFR 141)

1 Arsenic
2 Barium
3 Cadmium
4 Chromium
5 Lead
6 Mercury
7 Selenium
8 Silver

OTHERS

INDICATORS
1 pH
2 Percent solids

NITROGEN SERIES
1 Ammonia Nitrogen
2 Nitrate Nitrogen
3 Nitrite Nitrogen

CYANIDE
FLUORIDE
TOTAL PHOSPHORUS

TABLE 3

REMEDIAL INVESTIGATION SAMPLING AND ANALYSIS SEQUENCE

Site Category	Recon.	Phase I	Phase II	Contingency
Landfills	Geophysics	Cores - depth composites - screening & full priority pollutants & explosives residuals + ICP metals - Install wells-analyze indicators + metals.	Radial & depth cores and wells for priority pollutants & explosives residuals found in cores & AA metals.	
Surface	Geophysics - locate utilities	Surf. Soils - screening & full priority pollutants and explosive residuals + ICP metals.	Depth soils Radial soils - surf. & depth Runoff - water & sediments & depth profile	
Streams - Waters - Sediments		Upstream/downstream - screening & full priority pollutants & explosive residuals Surf. seds: 2 near shore, 1 near lake - screening & full priority pollutants + expl. + ICP metals	Surf seds - int + depth seds. - priority pollutants found + AA metals	
Ponds - Waters - Sediments - Groundwater		(Same rationale as streams) (Same rationale as streams) Upgradient/downgradient wells (2) - indicators	Depth profile on sediments priority pollutants + expl. found in waters or seds.	Additional wells
Lake - Waters - Sediments - Biota		5 sites; primary & secondary - Drinking Water stds. (None) Sample & freeze	10 sites + 5 use sites: anything found in Phase I 10 sites: parameters found in Phase I 5 sites: parameters found in Phase I	
Control Sites - Lake control - Soil & groundwater control - Clean area - Munitions area		(All analyses included at other sites)	Full scans	

*ICP: Metals analysis by Induced Coupled Plasma Spectrophotometry
AA: Metals analysis by Atomic Adsorption Spectrophotometry

TABLE 4
SUMMARY OF ANALYSES TO BE PERFORMED

	Task No. (WORK PLAN)	No. Samples Collected	No. For Screening (Phase I)	Full Analyses (Phase I)	Selected[3] Parameters (Phase II)	Field Analyses	Comments
2-B	Site Maps	--	--	--	--	--	1"=50' Scale with 1' contors
3-A	Geophysical Survey	6 sites				Terrain Conductivity	EM-31 Meter Used
	6 sites	--	--	--		Magnetometer	Proton Magneto-meter
3-B	Hyrdogeologic Investigations	9 wells: installed in Phase II -- Additional wells will be installed in Phase I.		--	--	Fire Sta. - 4 wells Acid Pond - 1 well Refuge control-1 well Munciation Control - 1 well Water Tower - 2 wells	2" ID PVC Casing and SS well screening
3-C	Groundwater Sampling and Analyses (1)	~~16~~ 24	--	--	See Table 7	Temp, pH and Spec. Conditions	Samples will be collected and Analyzed in Phase II
3-D	Soil Investigation	328	72(A) 192(B) 27(C) 15(D)	6-(F) 7(G) 9(H)	See Table 7		
3-E	Surface Water and Sediment Investigation	36	21(A) 5(E)				
		71	41(A) 7(D)	10(F) 3(G)	See Table 7		
3-F	Biota (2)	30	--	--	See Table 7	Length and Weight	Samples Frozen before shipping. Scheduled for analyses during Phase II.

Note:
The letters in parenthesis under screening and full analysis for Phase I indicate analysis sets (see Table 5).
(1) Sampling and Analyses of Ground Water scheduled for Phase II
(2) Fish samples obtained in Phase I, analyses scheduled for Phase II
(3) Specific details on parameters analysed

PARAMETER LIST FOR PHASE I (COMPLETED) ANALYSIS SETS

PARAMETERS		ANALYSIS SET A	B	C	D	E	F	G	H
1. Purgeable Priority Pollutants	-Screen	x	-	-	x	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x
2. Acid Extract. Priority Pollutants	-Screen	x	-	-	x	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x
3. Base/Neutral Extact. Prior. Poll.	-Screen	x	-	-	x	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x
4. Pesticide/PCB Priority Pollutants	-Screen	x	-	-	x	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x
5. PCB's		-	x	x	-	-	-	-	-
6. Metals - ICP Scan		x	-	-	x	-	-	-	-
- Prior. Poll. Metals by AA		-	-	-	-	-	-	-	x
- Mercury		x	-	-	x	-	-	-	-
7. EP Toxicity Metals		-	-	-	-	-	-	-	-
8. Cyanide		x	-	-	x	-	-	-	x
9. Indicators - pH (field)		x	-	x	x	-	-	-	x
- Specific Conductance (field)		x	-	x	x	-	-	-	x
- Total Organic Carbon		x	-	x	x	-	-	-	x
- Total Organic Halogen		x	-	x	x	-	-	-	x
10. Explosives Residues by HPLC		x	-	-	x	-	-	-	x
11. Nitrogen Series: TKN, NH3, NO3		x	-	x	x	-	-	-	x
12. PCDD/PCDF	-Screen	-	-	x	x	-	-	-	-
	-Full Anal.	-	-	-	-	-	-	x	x
13. Cation Exchange Capacity		-	-	x	-	-	x	x	-
14. Total Phosphorus		x	-	-	x	-	-	-	x
15. Primary & Secondary Drinking Water Stds.		-	-	-	-	x	-	-	-
16. Percent Solids (on soil/sed only)		x	x	x	x	-	x	x	x

NOTE: See Table 2C for list of compounds included within each Phase I parameter
Phase I Sampling and Analysis was completed in November 1985.
SETS F & G are full analysis of parameters screened in SETS A & D resply.
SET H is full analysis of selected samples instead of SET D

SUMMARY OF PHASE I SAMPLING AND ANALYSIS (Completed November 1985)

SITE NO.	SAMPLE TYPE	WATER NO.OF SAMPL	WATER ANAL. TYPE	SOILS NO.OF SAMPL	SOILS ANAL. TYPE	SEDIMENTS NO.OF SAMPL	SEDIMENTS ANAL. TYPE
3	AREA 11 SOUTH	0	-	3	A	1	A
				1	F	1	D
4	AREA 11 NORTH	0	-	1	D	1	A
						1	F
5	AREA 11 ACID POND	1	A	1	A	1	A
						1	F
7A	D AREA NORTH LAWN	0	-	16	A	0	-
				1	F		
11A	P AREA NORTH	0	-	4	A	4	A
						1	F
7	D AREA SOUTHEAST DRAINAGE	1	A	0	-	1	A
8	D AREA SOUTHWEST DRAINAGE	1	A	0	-	1	A
9	D AREA NORTHWEST DRAINAGE	1	A	0	-	1	A
10	WATERWORKS NORTH DRAINAGE	1	A	0	-	1	D
						1	G
11	P AREA SOUTHEAST DRAINAGE	1	A	0	-	1	A
						1	F
20	D AREA SOUTH	0	-	0	-	1	A
						1	F
12	AREA 14 IMPOUNDMENT	0	-	1	D	1	A
						1	G
13	AREA 14 CHANGE HOUSE SITE	0	-	6	A	0	-
14	AREA 14 SOLVENT STORAGE	2	A	0	-	2	A
						1	F
15	AREA 7 PLATING POND	1	A	0	-	1	A
16	AREA 7 INDUSTRIAL SITE	2	A	7	A	3	A
				2	D	1	F
				1	F		
				1	G		
17	JOB CORPS LANDFILL	2	A	5	A	0	-
				2	D		
				2	G		
18	AREA 13 LOADING PLATFORM	0	-	4	A	0	-
				1	F		

SUMMARY OF PHASE I SAMPLING AND ANALYSIS (Completed November 1985)

SITE NO.	SAMPLE TYPE	WATER NO.OF SAMPL	ANAL. TYPE	SOILS NO.OF SAMPL	ANAL. TYPE	SEDIMENTS NO.OF SAMPL	ANAL. TYPE
19	AREA 13 BUNKER 1-3	0	-	5	A	0	-
				1	F		
30	MUNITIONS CONTROL SITE	0	-	1	D	0	-
				1	G		
21	SOUTHEAST CORNER FIELD	0	-	4	A	0	-
				1	F		
22	OLD REFUGE SHOP	1	A	0	-	1	A
						1	F
24	PEPSI-WEST	1	A	0	-	1	A
						1	F
25	C.O.CREEK AT MARION LF	3	A	0	-	2	A
						1	D
						1	G
26	C.O.CREEK BELOW MARION STP	2	A	0	-	2	A
27	C.O.CREEK BELOW 157 DREDGE	1	A	0	-	1	D
28	WATER TOWER LANDFILL	0	-	11	A	0	-
				1	D		
				1	G		
29	FIRE STATION LANDFILL	0	-	5	A	0	-
				2	D		
				1	G		
32	AREA 9 LANDFILL	0	-	1	A	15	A
				8	B	3	D
				27	C		
				9	H		
33	AREA 9 BUILDING COMPLEX	0	-	184	B	0	-
				4	D		
35	AREA 9 EAST WATERWAY	0	-	0	-	1	A
						1	F
34	CRAB ORCHARD LAKE	5	E	0	-	0	-
31	REFUGE CONTROL SITE	0	-	1	D	0	-
				1	G		
	TOTAL NUMBER OF ANALYSES	26		328		61	415

SUMMARY BY ANALYSIS SETS OF PHASE I (Completed November 1985)

NO. OF ANALYSES	SCREENIN A	B	C	D	E	SUB-TOTAL	FULL ANALYSIS F	G	H	TOTAL
WATER	21	0	0	0	5	26	0	0	0	26
SOILS	72	192	27	15	0	306	6	7	9	328
SEDIMENTS	41	0	0	7	0	48	10	3	0	61
SUB-TOTAL	134	192	27	22	5	380	16	10	9	415
QA/QC - WATER	1	0	0	0	0	1	0	0	0	1
QA/QC - SOIL	12	31	4	6	0	53	1	2	2	58
QA/QC - SEDIMENT	7	0	0	1	0	8	2	1	0	11
QA/QC - BLANKS	9	0	0	1	0	10	0	2	1	13
QA/QC - TOTAL	29	31	4	8	0	72	3	5	3	83
TOTAL	163	223	31	30	5	452	19	15	12	498

PARAMETER LIST & UNIT COSTS FOR PHASE II ANALYSIS SETS

PARAMETERS	ANALYSIS SET															
	B	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W
1. CLP HSL Full Analysis	-	-	-	-	-	-	-	x	-	-	-	-	-	-	x	x
2. CLP HSL Volatiles	-	-	-	-	x	x	-	-	-	-	-	x	-	x	-	-
3. CLP HSL Base/Neut/Acids	-	x	x	-	x	-	-	-	-	-	-	-	-	x	-	-
4. Nitrosamines (CLP, soil)	-	-	-	-	-	-	-	-	-	x	-	-	-	-	-	-
5. Nitrosamines (low level)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	x	x
6. CLP HSL Pesticide/PCB	-	x	-	-	-	x	-	-	-	-	-	x	x	-	-	-
7. PCB's General	x	-	-	-	-	-	-	-	x	-	-	-	-	-	-	-
8. PCB's Low Level (water)	-	-	-	-	-	x	-	-	-	-	-	-	-	-	x	x
9. PCB's Semi-low (sediment)	-	x	-	-	-	-	-	-	-	x	-	-	-	-	-	-
10. Metals - CLP HSL	-	x	-	-	-	x	-	-	-	-	-	x	-	x	-	x
11. Metals - NIPDWR	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
12. Special - Mercury	-	-	-	x	-	-	-	-	-	-	-	-	x	-	-	-
- Cadmium	-	-	-	-	-	x	-	-	x	x	x	-	x	x	x	x
- Chromium	-	-	-	-	-	x	x	-	-	-	x	-	-	x	-	x
- Magnesium	-	-	-	-	-	-	-	x	-	-	-	-	-	-	-	-
- Lead	-	-	-	-	-	x	-	x	x	x	x	-	x	x	x	x
- Arsenic	-	-	-	-	-	x	-	x	-	-	-	-	-	x	x	x
- Copper	-	-	-	-	-	-	-	-	-	-	-	-	-	-	x	-
13. EP Toxicity - Cr	-	-	-	-	-	-	x	-	-	-	-	-	-	-	-	-
- Cd, Cr, Pb	-	-	-	-	-	-	-	-	-	-	x	-	-	-	-	-
14. Cyanide	-	x	x	x	-	-	-	-	-	-	x	x	-	x	-	x
15. Indicators - pH	-	x	x	-	x	x	x	x	-	x	x	x	-	x	x	x
- NH3, NO3, F	-	-	-	-	-	-	-	-	-	-	-	x	-	-	-	x
16. Explosives by HPLC	-	-	-	-	-	-	-	-	-	x	-	-	-	-	x	x
17. Lipids	-	-	-	-	-	-	-	-	-	-	-	-	x	-	-	-
18. PCDD/PCDF	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
19. Total Phosphorus	-	-	-	-	-	x	-	-	-	-	-	-	-	-	-	-
20. Grain Size	-	-	-	-	-	-	-	-	-	-	-	x	-	-	-	x
21. Percent Solids (soil/sed	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x

NOTE: See Table 2D for list of compounds included within each parameter
See Table 7C of SOP for detection levels; Table 10 of QAPP for analytical procedures
Nitrosamines in water & well samples will be analyzed using a lower detection level
Well water metals analyses include unfiltered and filtered

PARAMETER LIST & UNIT COSTS FOR PHASE II ANALYSIS SETS

PARAMETERS	ANALYSIS SET (contd.)														
	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ	AK	AL
1. CLP HSL Full Analysis	x	-	-	-	-	-	x	-	-	x	x	x	-	x	-
2. CLP HSL Volatiles	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
3. CLP HSL Base/Neut/Acids	-	x	x	-	-	-	-	-	-	-	-	-	-	-	-
4. Nitrosamines (CLP, soil)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
5. Nitrosamines (low level)	x	-	-	-	-	-	-	-	-	x	-	-	-	x	x
6. CLP HSL Pesticide/PCB	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
7. PCB's General	-	-	-	-	x	-	-	-	-	-	-	-	-	-	-
8. PCB's Low Level (water)	x	-	-	-	-	-	-	-	-	x	-	-	-	x	x
9. PCB's Semi-low (sediment)	-	-	-	-	-	-	x	-	x	-	x	-	-	-	-
10. Metals - CLP HSL	x	-	-	-	-	-	-	-	-	x	x	x	-	-	-
11. Metals - NIPDWR	-	-	-	-	-	-	-	-	-	-	-	-	-	x	-
12. Special - Mercury	-	-	-	-	-	-	-	x	-	-	-	-	x	-	x
- Cadmium	x	-	x	-	-	-	-	-	-	x	-	-	-	-	x
- Chromium	x	-	x	-	-	-	-	x	-	x	-	-	-	-	x
- Magnesium	-	-	-	-	x	x	-	-	-	-	-	-	-	-	-
- Lead	x	-	-	-	x	x	x	x	x	x	-	-	-	-	x
- Arsenic	x	-	-	-	x	-	-	-	-	x	-	-	-	-	x
- Copper	-	-	-	-	x	-	-	-	-	-	-	-	-	-	-
13. EP Toxicity - Cr	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
- Cd, Cr, Pb	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
14. Cyanide	x	-	x	x	x	-	-	-	-	x	x	-	x	x	x
15. Indicators - pH	x	-	-	-	-	x	x	-	-	x	x	-	-	x	x
- NH3, NO3, F	x	-	-	-	-	-	-	-	-	x	-	-	-	-	x
16. Explosives by HPLC	-	-	-	-	-	-	-	-	-	-	x	-	-	-	-
17. Lipids	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
18. PCDD/PCDF	-	-	-	-	-	-	-	-	-	-	x	-	-	-	-
19. Total Phosphorus	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
20. Grain Size	-	-	-	-	-	-	-	-	-	x	-	-	-	-	-
21. Percent Solids (soil/sed	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x

NOTE: See Table 2D for list of compounds included within each parameter
See Table 7C of SOP for detection levels; Table 10 of QAPP for analytical procedures
Nitrosamines in water & well samples will be analyzed using a lower detection level
Well water metals analyses include unfiltered and filtered

PHASE II SAMPLING AND ANALYSIS SUMMARY BY SITES

SITE NO.	SAMPLE TYPE	WATER NO.OF SAMPL	WATER ANAL. TYPE	WELL NO.OF SAMPL	WELL ANAL. TYPE	SOILS NO.OF SAMPL	SOILS ANAL. TYPE	SEDIMENTS NO.OF SAMPL	SEDIMENTS ANAL. TYPE	BIOTA NO.OF SAMPL	BIOTA ANAL. TYPE
	NOTE: * indicates re-sampling/re-analysis of Phase I samples										
3	AREA 11 SOUTH	No Phase II sampling and/or analysis									
4	AREA 11 NORTH	No Phase II sampling and/or analysis									
5	AREA 11 ACID POND	No Phase II sampling and/or analysis									
7A	D AREA NORTH LAWN	0	-	0	-	6	AJ *	0	-	0	-
11A	P AREA NORTH	0	-	0	-	1	AJ *	0	-	0	-
7	D AREA SOUTHEAST DRAINAGE	0	-	0	-	0	-	1	AJ *	0	-
8	D AREA SOUTHWEST DRAINAGE	No Phase II sampling and/or analysis									
9	D AREA NORTHWEST DRAINAGE	0	-	0	-	0	-	1	K *	0	-
10	WATERWORKS NORTH DRAINAGE	1	J	0	-	0	-	5	J	0	-
								1	AJ *		
11	P AREA SOUTHEAST DRAINAGE	1	K *	0	-	0	-	1	AJ *	0	-
20	D AREA SOUTH	1	K *	0	-	0	-	0	-	0	-
12	AREA 14 IMPOUNDMENT	No Phase II sampling and/or analysis									
13	AREA 14 CHANGE HOUSE SITE	No Phase II sampling and/or analysis									
14	AREA 14 SOLVENT STORAGE	1	L	0	-	0	-	1	L	0	-
15	AREA 7 PLATING POND	0	-	1	M	0	-	1	N	0	-
16	AREA 7 INDUSTRIAL SITE	1	O	0	-	0	-	1	O	0	-
17	JOB CORPS LANDFILL	2	V	5	W	35	P	6	Q	0	-
						12	Q				
18	AREA 13 LOADING PLATFORM	No Phase II sampling and/or analysis									
19	AREA 13 BUNKER 1-3	0	-	0	-	1	AJ *	0	-	0	-
30	MUNITIONS CONTROL SITE	0	-	1	X	1	Y	0	-	0	-
21	SOUTHEAST CORNER FIELD	0	-	0	-	1	AJ *	0	-	0	-
22	OLD REFUGE SHOP	0	-	1	U	1	Z	3	R	0	-
								1	Z		

PHASE II SAMPLING AND ANALYSIS SUMMARY BY SITES

SITE NO.	SAMPLE TYPE	WATER NO.OF SAMPL	ANAL. TYPE	WELL NO.OF SAMPL	ANAL. TYPE	SOILS NO.OF SAMPL	ANAL. TYPE	SEDIMENTS NO.OF SAMPL	ANAL. TYPE	BIOTA NO.OF SAMPL	ANAL. TYPE
	NOTE: * indicates re-sampling/re-analysis of Phase I samples										
24	PEPSI-WEST	0	-	0	-	0	-	1	AJ *	0	-
25	C.O.CREEK AT MARION LF	0	-	0	-	0	-	1	AA *	0	-
26	C.O.CREEK BELOW MARION STP	No Phase II sampling and/or analysis									
27	C.O.CREEK BELOW 157 DREDGE	No Phase II sampling and/or analysis									
28	WATER TOWER LANDFILL	0	-	4	S	4	AB	0	-	0	-
29	FIRE STATION LANDFILL	0	-	5	S	13	AC	0	-	0	-
32	AREA 9 LANDFILL	0	-	5	AG	24	AE	37	AF	0	-
								5	AD		
33	AREA 9 BUILDING COMPLEX	0	-	3	X	148	B	0	-	0	-
						3	AI				
35	AREA 9 EAST WATERWAY	No Phase II sampling and/or analysis									
34	CRAB ORCHARD LAKE	10	AL	0	-	0	-	8	I	30	T
		5	AK					2	AH		
31	REFUGE CONTROL SITE	0	-	1	X	1	Y	0	-	0	-
	TOTAL NUMBER OF ANALYSES	22		26		251		76		30	405

PHASE II SAMPLING AND ANALYSIS SUMMARY BY SETS

ANALYSIS SET

NO. OF ANALYSES	B	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y
WATER	0	0	1	2	1	0	0	1	0	0	0	0	0	0	2	0	0	0
WELLS	0	0	0	0	0	1	0	0	0	0	0	9	0	1	0	5	5	0
SOILS	148	0	0	0	0	0	0	0	35	12	0	0	0	0	0	0	0	2
SEDIMENTS	0	8	5	1	1	0	1	1	0	6	3	0	0	0	0	0	0	0
BIOTA	0	0	0	0	0	0	0	0	0	0	0	0	30	0	0	0	0	0
SUB-TOTAL	148	8	6	3	2	1	1	2	35	18	3	9	30	1	2	5	5	2
QA/QC - WATER	0	0	0	0	1	0	0	0	0	0	0	0	0	0	1	0	0	0
QA/QC - WELL	0	0	0	0	0	0	0	0	0	0	0	2	0	0	0	1	1	0
QA/QC - SOIL	23	0	0	0	0	0	0	0	5	2	0	0	0	0	0	0	0	1
QA/QC - SEDIMENT	0	2	1	0	0	0	0	0	0	1	1	0	0	0	0	0	0	0
QA/QC - BLANKS	2	0	1	0	0	0	0	0	1	1	0	0	0	0	1	0	1	1
QA/QC - TOTAL	25	2	2	0	1	0	0	0	6	4	1	2	0	0	2	1	2	2
TOTAL	173	10	8	3	3	1	1	2	41	22	4	11	30	1	4	6	7	4

SAMPLING AND ANALYSIS SUMMARY BY SETS

ANALYSIS SET (Cont'd)

NO. OF ANALYSES	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ	AK	AL	TOTAL
WATER	0	0	0	0	0	0	0	0	0	0	0	5	10	22
WELLS	0	0	0	0	0	0	0	5	0	0	0	0	0	26
SOILS	1	0	4	13	0	24	0	0	0	3	9	0	0	251
SEDIMENTS	1	1	0	0	5	0	37	0	2	0	4	0	0	76
BIOTA	0	0	0	0	0	0	0	0	0	0	0	0	0	30
SUB-TOTAL	2	1	4	13	5	24	37	5	2	3	13	5	10	405
QA/QC - WATER	0	0	0	0	0	0	0	0	0	0	0	2	2	6
QA/QC - WELL	0	0	0	0	0	0	0	1	0	0	0	0	0	5
QA/QC - SOIL	1	0	1	2	0	3	0	0	0	1	2	0	0	41
QA/QC - SEDIMENT	0	0	0	0	1	0	6	0	1	0	1	0	0	14
QA/QC - BLANKS	0	0	1	1	1	0	1	1	0	0	0	0	1	14
QA/QC - TOTAL	1	0	2	3	2	3	7	2	1	1	3	2	3	80
TOTAL	3	1	6	16	7	27	44	7	3	4	16	7	13	485

Does not
include 6 reanalyses
for arsenic.

PHASE II SAMPLING

SITE # 3
AREA 11 SOUTH

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING

SITE # 4
AREA 11 NORTH

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING

SITE # 5
AREA 11 ACID POND

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING

SITE # 7A

D AREA NORTH LAWN

PARAMETERS	NUMBER OF SAMPLES SOILS
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	6
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	6

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. No Phase II sampling
6. The six soils are Phase I samples for Hg re-analysis

PHASE II SAMPLING

SITE # 11A

P AREA NORTH

PARAMETERS	NUMBER OF SAMPLES SOILS
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. No Phase II sampling
6. The one soil is Phase I sample for Hg re-analysis

PHASE II SAMPLING

SITE # 7

D AREA SOUTHEAST DRAINAGE

PARAMETERS	NUMBER OF SAMPLES SEDIMENT
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. No Phase II sampling
6. One sediment is Phase I sample for Hg re-analysis

PHASE II SAMPLING

SITE # 8

D AREA SOUTHWEST DRAINAGE

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	NO PHASE II
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING
SITE # 9
P AREA NORTHWEST DRAINAGE

PARAMETERS	NUMBER OF SAMPLES SEDIMENT
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	1
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. One sediment is re-sample and analysis for Hg and CN

PHASE II SAMPLING
SITE # 10
WATERWORKS NORTH DRAINAGE

PARAMETERS	NUMBER OF SAMPLES WATER	SEDIMENT
1. CLP HSL Full Analysis		
2. CLP HSL Volatiles		
3. CLP HSL Base/Neut/Acids	1	5 ,-
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL		
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		- ,1
- Cadmium		
- Chromium		
- Magnesium		
- Lead		
- Arsenic		
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	1	5 ,-
15. Indicators - pH	1	5 ,-
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		5 ,-

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Total of 6 sediment samples: five sediment for Phase II; one Phase I sediment sample re-analyzed for Hg

PHASE II SAMPLING
SITE # 11
P AREA SOUTHEAST DRAINAGE

PARAMETERS	NUMBER OF SAMPLES WATER	SEDIMENT
1. CLP HSL Full Analysis		
2. CLP HSL Volatiles		
3. CLP HSL Base/Neut/Acids		
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL		
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury	.1	1
- Cadmium		
- Chromium		
- Magnesium		
- Lead		
- Arsenic		
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	1	
15. Indicators - pH	1	
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. One Phase I sediment sample re-analyzed for Hg
6. One water for re-sampling and analysis

PHASE II SAMPLING
SITE # 20
D AREA SOUTH

PARAMETERS	NUMBER OF SAMPLES WATER
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	1
15. Indicators - pH	1
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Water sample is re-analysis; If no water is available, will use sediment leachate

PHASE II SAMPLING

SITE # 12
AREA 14 IMPOUNDMENT

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	NO PHASE II
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Small mammals collected

PHASE II SAMPLING

SITE # 13

AREA 14 CHANGE HOUSE SITE

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING
SITE # 14
AREA 14 SOLVENT STORAGE

PARAMETERS	NUMBER OF SAMPLES WATER	SEDIMENT
1. CLP HSL Full Analysis		
2. CLP HSL Volatiles	1	1
3. CLP HSL Base/Neut/Acids	1	1
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL		
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium		
- Chromium		
- Magnesium		
- Lead		
- Arsenic		
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide		
15. Indicators - pH	1	1
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. EPA to request SPCC inspctn.

PHASE II SAMPLING
SITE # 15
AREA 7 PLATING POND

PARAMETERS	NUMBER OF SAMPLES WELL	SEDIMENT
1. CLP HSL Full Analysis		
2. CLP HSL Volatiles	1	
3. CLP HSL Base/Neut/Acids		
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB	1	
7. PCB's (general)		
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL	1	
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium	1	
- Chromium	1	1
- Magnesium		
- Lead	1	
- Arsenic	1	
- Copper		
13. EP Toxicity - Cr		1
- Cd, Cr, Pb		
14. Cyanide		
15. Indicators - pH	1	1
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus	1	
20. Grain Size		
21. Percent Solids (soil/sed)		1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Two piezometers for GW level
6. Field permeability in well

PHASE II SAMPLING
SITE # 16
AREA 7 INDUSTRIAL SITE

PARAMETERS	NUMBER OF SAMPLES WATER	SEDIMENT
1. CLP HSL Full Analysis	1	1
2. CLP HSL Volatiles		
3. CLP HSL Base/Neut/Acids		
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL		
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium		
- Chromium		
- Magnesium	1	1
- Lead	1	1
- Arsenic	1	1
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide		
15. Indicators - pH	1	1
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING
SITE # 17
JOB CORPS LANDFILL

PARAMETERS	NUMBER OF SAMPLES WATER	WELL	SOILS	SEDIMENT
1. CLP HSL Full Analysis	2	4 ,1		
2. CLP HSL Volatiles				
3. CLP HSL Base/Neut/Acids				
4. Nitrosamines (CLP, soil)			- ,12	
5. Nitrosamines (low, water)	2	4 ,1		
6. CLP HSL Pesticide/PCB				
7. PCB's (general)			35 ,-	
8. PCB's (low level, water)	2	4 ,1		
9. PCB's (semi low, sediment)			- ,12	6
10. Metals - CLP HSL		4 ,1		
11. Metals - NIPDWR(40CFR141)				
12. Special - Mercury			1	
- Cadmium	2	4 ,1	35 ,12	6
- Chromium		4 ,1		
- Magnesium				
- Lead	2	4 ,1	35 ,12	6
- Arsenic	2	4 ,1		
- Copper	2			
13. EP Toxicity - Cr				
- Cd, Cr, Pb				
14. Cyanide		4 ,1		
15. Indicators - pH	2	4 ,1	- ,12	6
- NH3, NO3, F		4 ,1		
16. Explosives by HPLC	2	4 ,1	- ,12	6
17. Lipids				
18. PCDD/PCDF				
19. Total Phosphorus				
20. Grain Size		4 ,1		
21. Percent Solids (soil/sed)				6

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. 4 shallow and one deep well,
6. 35 surface and 12 core soils,
7. Field permeability in wells
8. One Phase I soil sample will be re-analyzed for Hg
9. Small mammals for liver

This is not the only site with nitrosamines nor is it the highest.

PHASE II SAMPLING

SITE # 18
AREA 13 LOADING PLATFORM

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Small mammals for liver examination

PHASE II SAMPLING

SITE # 19
AREA 13 BUNKER 1-3

PARAMETERS	NUMBER OF SAMPLES SOILS
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. No Phase II sampling
6. The one soil is Phase I sample for Hg re-analysis

PHASE II SAMPLING
SITE # 30
MUNITIONS CONTROL SITE

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS
1. CLP HSL Full Analysis	1	
2. CLP HSL Volatiles		
3. CLP HSL Base/Neut/Acids		1
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)	1	
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		
8. PCB's (low level, water)	1	
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL	1	
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium	1	
- Chromium	1	
- Magnesium		
- Lead	1	
- Arsenic	1	
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	1	
15. Indicators - pH	1	
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. One surface soil

PHASE II SAMPLING

SITE # 21

SOUTHEAST CORNER FIELD

PARAMETERS	NUMBER OF SAMPLES SOILS
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. No Phase II sampling
6. The one soil is Phase I sample for Hg re-analysis

PHASE II SAMPLING

SITE # 22

OLD REFUGE SHOP

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS	SEDIMENT
1. CLP HSL Full Analysis			
2. CLP HSL Volatiles	1		
3. CLP HSL Base/Neut/Acids	1	1	1 ,-
4. Nitrosamines (CLP, soil)			
5. Nitrosamines (low, water)			
6. CLP HSL Pesticide/PCB			
7. PCB's (general)			
8. PCB's (low level, water)			
9. PCB's (semi low, sediment)			
10. Metals - CLP HSL	1		
11. Metals - NIPDWR(40CFR141)			
12. Special - Mercury			
- Cadmium	1	1	1 ,3
- Chromium	1	1	1 ,3
- Magnesium			
- Lead	1		- ,3
- Arsenic	1		
- Copper			
13. EP Toxicity - Cr			
- Cd, Cr, Pb			- ,3
14. Cyanide	1	1	1 ,3
15. Indicators - pH	1		- ,3
- NH3, NO3, F			
16. Explosives by HPLC			
17. Lipids			
18. PCDD/PCDF			
19. Total Phosphorus			
20. Grain Size			
21. Percent Solids (soil/sed)		1	1 ,3

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Total of 4 sediment samples
6. Field permeability in well

PHASE II SAMPLING

SITE # 24
PEPSI-WEST

PARAMETERS	NUMBER OF SAMPLES SEDIMENT
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	1
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. No Phase II sampling
6. One sediment is Phase I sample for Hg re-analysis

PHASE II SAMPLING
SITE # 25
C.O. CREEK AT MARION LF

PARAMETERS	NUMBER OF SAMPLES SEDIMENT
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	1
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Sediment re-sampled for CN

PHASE II SAMPLING

SITE # 26

C.O. CREEK BELOW MARION STP

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING

SITE # 27

C.O. CREEK BELOW 157 DREDGE

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING
SITE # 28
WATER TOWER LANDFILL

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS
1. CLP HSL Full Analysis		
2. CLP HSL Volatiles	3 , 1	
3. CLP HSL Base/Neut/Acids		
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB	3 , 1	
7. PCB's (general)		4
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL	3 , 1	
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium		
- Chromium		
- Magnesium		4
- Lead		4
- Arsenic		4
- Copper		4
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	3 , 1	4
15. Indicators - pH	3 , 1	
- NH3, NO3, F	3 , 1	
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size	3 , 1	
21. Percent Solids (soil/sed)		4

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. 4 soils from two test pits; safety plan in QAPP (Rev.3)
6. 3 shallow and 1 deep well, 35 surface and 12 core soils,
7. Field permeability in wells
8. One soil sample re-run for CN

PHASE II SAMPLING
SITE # 29
FIRE STATION LANDFILL

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS
1. CLP HSL Full Analysis		
2. CLP HSL Volatiles	4 , 1	
3. CLP HSL Base/Neut/Acids		
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)		
6. CLP HSL Pesticide/PCB	4 , 1	
7. PCB's (general)		
8. PCB's (low level, water)		
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL	4 , 1	
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium		
- Chromium		
- Magnesium		13
- Lead		13
- Arsenic		
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	4 , 1	
15. Indicators - pH	4 , 1	
- NH3, NO3, F	4 , 1	
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size	4 , 1	
21. Percent Solids (soil/sed)		13

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. 4 shallow and 1 deep well,
6. Field permeability in wells
7. Six Phase I soil samples will be re-analyzed for Hg

PHASE II SAMPLING
SITE # 32
AREA 9 LANDFILL

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS	SEDIMENT
1. CLP HSL Full Analysis	4 ,1		- ,5
2. CLP HSL Volatiles			
3. CLP HSL Base/Neut/Acids			
4. Nitrosamines (CLP, soil)			
5. Nitrosamines (low, water)	4 ,1		
6. CLP HSL Pesticide/PCB			
7. PCB's (general)			
8. PCB's (low level, water)	4 ,1		
9. PCB's (semi low, sediment)			37 ,5
10. Metals - CLP HSL	4 ,1		
11. Metals - NIPDWR(40CFR141)			
12. Special - Mercury		24 ,9	
- Cadmium	4 ,1		
- Chromium	4 ,1	24 ,9	
- Magnesium			
- Lead	4 ,1	24 ,9	37 ,5
- Arsenic	4 ,1		
- Copper			
13. EP Toxicity - Cr			
- Cd, Cr, Pb			
14. Cyanide	4 ,1		
15. Indicators - pH	4 ,1		
- NH3, NO3, F	4 ,1		
16. Explosives by HPLC			
17. Lipids			
18. PCDD/PCDF			
19. Total Phosphorus			
20. Grain Size	4 ,1		
21. Percent Solids (soil/sed)		24 ,9	37 ,5

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. 4 shallow and 1 deep well
6. Total of 42 sediment samples
7. 24 surface soils from Phase I
8. 9 Phase I bottom comp. soils re-analyzed for Hg, Cr & Pb
9. Field permeability in wells

OK

PHASE II SAMPLING
SITE # 33
AREA 9 BUILDING COMPLEX

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS
1. CLP HSL Full Analysis	3	- ,3
2. CLP HSL Volatiles		
3. CLP HSL Base/Neut/Acids		
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)	3	
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		148 ,-
8. PCB's (low level, water)	3	
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL	3	- ,3
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium	3	
- Chromium	3	
- Magnesium		
- Lead	3	
- Arsenic	3	
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	3	
15. Indicators - pH	3	
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		148 ,3

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. Field permeability in wells
6. Total 151 sediment samples

PHASE II SAMPLING

SITE # 35
AREA 9 EAST WATERWAY

PARAMETERS	NUMBER OF SAMPLES
1. CLP HSL Full Analysis	
2. CLP HSL Volatiles	
3. CLP HSL Base/Neut/Acids	
4. Nitrosamines (CLP, soil)	
5. Nitrosamines (low, water)	
6. CLP HSL Pesticide/PCB	
7. PCB's (general)	
8. PCB's (low level, water)	
9. PCB's (semi low, sediment)	NO PHASE II
10. Metals - CLP HSL	
11. Metals - NIPDWR(40CFR141)	
12. Special - Mercury	
- Cadmium	
- Chromium	
- Magnesium	
- Lead	
- Arsenic	
- Copper	
13. EP Toxicity - Cr	
- Cd, Cr, Pb	
14. Cyanide	
15. Indicators - pH	
- NH3, NO3, F	
16. Explosives by HPLC	
17. Lipids	
18. PCDD/PCDF	
19. Total Phosphorus	
20. Grain Size	
21. Percent Solids (soil/sed)	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

PHASE II SAMPLING
SITE # 34
CRAB ORCHARD LAKE

PARAMETERS	NUMBER OF SAMPLES WATER	SEDIMENT	BIOTA
1. CLP HSL Full Analysis	- ,5	- ,2	
2. CLP HSL Volatiles			
3. CLP HSL Base/Neut/Acids		8 ,-	
4. Nitrosamines (CLP, soil)			
5. Nitrosamines (low, water)	10 ,5		
6. CLP HSL Pesticide/PCB		8 ,-	30
7. PCB's (general)			
8. PCB's (low level, water)	10 ,5		
9. PCB's (semi low, sediment)		8 ,2	
10. Metals - CLP HSL		8 ,2	
11. Metals - NIPDWR(40CFR141)	- ,5		
12. Special - Mercury			30
- Cadmium	10 ,-		30
- Chromium	10 ,-		
- Magnesium			
- Lead	10 ,-		30
- Arsenic	10 ,-		
- Copper			
13. EP Toxicity - Cr			
- Cd, Cr, Pb			
14. Cyanide	10 ,5	8 ,2	
15. Indicators - pH	10 ,5	8 ,2	
- NH3, NO3, F	10 ,-		
16. Explosives by HPLC		- ,2	
17. Lipids			30
18. PCDD/PCDF		- ,2	
19. Total Phosphorus			
20. Grain Size			
21. Percent Solids (soil/sed)		8 ,2	

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. 10 vertical water column & 5 drinking water sources
6. Total of 6 sediment samples
7. See Table 7B for fish species and duplicates

PHASE II SAMPLING
SITE # 31
REFUGE CONTROL SITE

PARAMETERS	NUMBER OF SAMPLES WELL	SOILS
1. CLP HSL Full Analysis	1	
2. CLP HSL Volatiles		
3. CLP HSL Base/Neut/Acids		1
4. Nitrosamines (CLP, soil)		
5. Nitrosamines (low, water)	1	
6. CLP HSL Pesticide/PCB		
7. PCB's (general)		
8. PCB's (low level, water)	1	
9. PCB's (semi low, sediment)		
10. Metals - CLP HSL	1	
11. Metals - NIPDWR(40CFR141)		
12. Special - Mercury		
- Cadmium	1	
- Chromium	1	
- Magnesium		
- Lead	1	
- Arsenic	1	
- Copper		
13. EP Toxicity - Cr		
- Cd, Cr, Pb		
14. Cyanide	1	
15. Indicators - pH	1	
- NH3, NO3, F		
16. Explosives by HPLC		
17. Lipids		
18. PCDD/PCDF		
19. Total Phosphorus		
20. Grain Size		
21. Percent Solids (soil/sed)		1

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes
5. One surface soil

PHASE II SAMPLING
REFUGE TOTAL
ALL SITES

PARAMETERS	WATR	WELL	SOIL	SED.	BIOTA	TOTAL
1. CLP HSL Full Analysis	8	15	3	8	0	34
2. CLP HSL Volatiles	1	11	0	1	0	13
3. CLP HSL Base/Neut/Acids	2	1	3	15	0	21
4. Nitrosamines (CLP, soil)	0	0	12	0	0	12
5. Nitrosamines (low, water)	17	15	0	0	0	32
6. CLP HSL Pesticide/PCB	0	10	0	8	30	48
7. PCB's (general)	0	0	187	0	0	187
8. PCB's (low level, water)	17	15	0	0	0	32
9. PCB's (semi low, sediment)	0	0	12	58	0	70
10. Metals - CLP HSL	0	26	3	10	0	39
11. Metals - NIPDWR(40CFR141)	5	0	0	0	0	5
12. Special - Mercury	2	0	43	5	30	80
- Cadmium	12	17	48	10	30	117
- Chromium	10	17	34	5	0	66
- Magnesium	1	0	17	1	0	19
- Lead	13	17	97	52	30	209
- Arsenic	13	17	4	1	0	35
- Copper	2	0	4	0	0	6
13. EP Toxicity - Cr	0	0	0	1	0	1
- Cd, Cr, Pb	0	0	0	3	0	3
14. Cyanide	18	25	5	21	0	69
15. Indicators - pH	22	26	12	27	0	87
- NH3, NO3, F	10	19	0	0	0	29
16. Explosives by HPLC	2	5	12	8	0	27
17. Lipids	0	0	0	0	30	30
18. PCDD/PCDF	0	0	0	2	0	2
19. Total Phosphorus	0	1	0	0	0	1
20. Grain Size	0	19	0	0	0	19
21. Percent Solids (soil/sed)	0	0	213	75	0	288
						1581

NOTE:
1. Detection levels in Table 7C
2. See analytical procedures in Table 10 of QAPP (Sept. 1986)
3. See Table 4 for compounds included in parameters
4. See Table 6, pages 3 & 4 for field duplicates and spikes

CONWR Table 7B Oct. 20, 1986

SUMMARY OF BIOTA SAMPLING FOR PHASE II ANALYSIS

CRAB ORCHARD LAKE SITE No.		NUMBER OF FISH PER COMPOSITE	SPECIES
1		5	Carp
		5	Bass
	*	5	Bass
		5	Bullhead
	**	5	Bullhead
		2	Catfish
2	**	5	Carp
	*	5	Carp
		5	Bass
		5	Bullhead
	*	5	Bullhead
		5	Catfish
3		5	Carp
	*	5	Carp
	**	5	Bass
		5	Bullhead
	*	5	Bullhead
4	**	5	Carp
		5	Bass
	*	5	Bass
		5	Bullhead
	*	5	Bullhead
		4	Catfish
Lake Control		5	Carp
	*	5	Carp
	**	3	Bass
	*	5	Bass
		5	Bullhead
	*	5	Bullhead
		3	Catfish

NOTES: 1. Procedures for fish preparation & analysis will be submitted separately
2. (*) = Duplicate composites for OB&G analysis
3. (**) = Duplicate composites for FWS analysis

PHASE II ANALYSIS
NO OF ANALYSIS AND DETECTION LEVELS

PARAMETERS	WATER & WELL			SOIL & SEDIMENTS		
	No. of Samples	Detection Level	Pg. no. in Table 10	No. of Analysis	Detection Level	Pg. no. in Table 10
1. CLP HSL Full Analysis	23	.05-50 ppb	1	11	10-1600 ppb	13
2. CLP HSL Volatiles	12	10 ppb	1	1	10 ppb	13
3. CLP HSL Base/Neut/Acids	3	10-50 ppb	2	18	330-1600 ppb	14
4. Nitrosamines (CLP, soil)				12	330 ppb	16
5. Nitrosamines (low, water)	32	0.1-0.8 ppb	4			
6. CLP HSL Pesticide/PCB	10	.05-1 ppb	5	8	80-160 ppb	17
7. PCB's General				187	500 ppb	18
8. PCB's Low Level (water)	25	5 ppt	6			
9. PCB's Semi-low (sediment)				22	40 ppb	19
10. Metals - CLP HSL	26	5-5000 ppb	7	13	0.1-80 ppm	20
11. Metals - NIPDWR	5	0.2-1000 ppb	9			
12. Sp. - Mercury	2	0.2 ppb	10	48 *	20 ppb	21
- Cadmium - Flame				58	500 ppb	21
- Furnace	24	1 ppb	10			
- Chromium - Flame				39	5000 ppb	21
- Furnace	22	1 ppb	10			
- Magnesium - Flame	1	10 ppb	10	18	1000 ppb	21
- Lead - Flame				149	10000 ppb	21
- Furnace	25	0.2 ppb	10			
- Arsenic - Furnace	25	5 ppb	10	5	100 ppb	21
- Copper - Flame				4	2000 ppb	21
- Furnace	2	1.0 ppb	10			
13. EP Toxicity - Cr				1	1000 ppb	
- Cd, Cr, Pb				3	100 ppb	
14. Cyanide	43	50 ppb	11	26 *	5000 ppb	22
15. Indicators - pH	48	-	11	39	--	22
- NH3, NO3, F	29	10,10,50 ppb	11			
16. Explosives by HPLC	7	0.4-2.0 ppb	12	20	500 ppb	24
17. Lipids						
18. PCDD/PCDF				2	0.02-0.2 ppb	23
19. Total Phosphorus	1	10 ppb	11	2	1000 ppb	22
21. Percent Solids (soil/sed)				285	0.1%	22

NOTE: 1. See Table 4 for list of compounds included within each parameter
2. See Table 10 of QAPP for analytical procedures
3. (*) Phase I re-sampling is included above
4. (**) Procedures for fish analysis will be submitted separately

TABLE 8
FUNCTIONAL ACTIVITIES

Task/Activity	Responsible Company	Where Performed
Task 1 - Description of Current Situation	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
Task 2 - Remedial Investigation Support		
Support - A - Site Visit	O'Brien & Gere Engineers, Inc.	On-Site
B - Site Maps	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
Task 3 - Site Investigations		
A - Geophysical Surveys	O'Brien & Gere Engineers, Inc.	On-Site
B - Hydrogeologic Investigations	O'Brien & Gere Engineers, Inc.	On-Site
- Installation of Monitoring Wells	Professional Service Industries, Inc. with O'Brien & Gere Engineers, Inc. Supervising	On-Site
C - Groundwater: Sampling	O'Brien & Gere Engineers, Inc.	On-Site
Analyses	O'Brien & Gere Laboratories, Inc.	Laboratory - Syracuse, New York
	Rocky Mountain	Denver, Colorado
	Environmental Testing & Certification (ETC)	Laboratory - Edison, New York
D - Soil Investigation: Sampling	O'Brien & Gere Engineers, Inc.	On-Site
Analyses	O'Brien & Gere Laboratories, Inc.	Laboratory, Syracuse, New York
	Roy F. Weston, Inc.	Laboratory, West Chester, Penn
E - Surface Water & Sediment Investigation: Sampling	O'Brien & Gere Engineers, Inc.	On-Site
	O'Brien & Gere Laboratories, Inc.	Laboratory, Syracuse, New York
Analyses	Roy F. Weston, Inc.	Laboratory, West Chester, Penn
	Rocky Mountain	Denver, Colorado
	Environmental Testing & Certification (ETC)	Laboratory, Edison, New York
F - Biota: Sampling	O'Brien & Gere Engineers, Inc.	On-Site
Analyses	O'Brien & Gere Engineers, Inc.	Laboratory, Syracuse, New York
Task 4 - Preliminary Remedial Technologies	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
Task 5 - Site Investigations Analysis	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
Task 6 - Final Report	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
Task 7 - Community Relations	Fish and Wildlife Service	On-Site
Task 8 - Additional Requirements	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York

ANALYTICAL RESPONSIBILITIES

PARAMETERS	OB&G	ETC	Rocky Mt	Weston
1. CLP HSL Full Analysis		W/S		
2. CLP HSL Volatiles		W/S		
3. CLP HSL Base/Neut/Acids		W/S		
4. Nitrosamines (CLP, soil)		W/S		
5. Nitrosamines (low level, water)		W/S		
6. CLP HSL Pesticide/PCB		W/S		
7. PCB's (general, soil)	S			
8. PCB's (low level, water)	W			
9. PCB's (semi-low, sediment)	S			
10. Metals - CLP HSL			W/S	
11. Metals - NIPDWR (water)			W	
12. Special - Mercury			W/S	
- Cadmium	S		W/S	
- Chromium			W/S	
- Lead	S		W/S	
- Arsenic			W/S	
- Copper			W/S	
- Magnesium	S		W/S	
13. EP Toxicity - Cr	S			
- Cd, Cr, Pb	S			
14. Cyanide	S/W			
15. Indicators - pH	S/W			
- NH3, NO3, F	S/W			
16. Explosives by HPLC				W/S
17. Lipids (biota) *				
18. PCDD/PCDF (sediment)		S		
19. Total Phosphorus	S/W			
20. Grain Size	S			
21. Percent Solids (soil/sed)	S/W	S/W	W/S	S/W

NOTES: 1. OBG - O'Brien & Gere Laboratories, Syracuse, NY
ETC - Environmental Testing & Certification, Edison, NJ
Rocky Mt. - Rocky Mountain Labs, Denver, CO
Weston - Roy F. Weston, Inc., West Chester, PA
* OBG - metals in soils if only Cd, Pd & Mg are scheduled

2. W/S/B/ denote: W - water/well
S - soil/sediment
B - biota

3. Laboratories analyzing biota will be included separately

TABLE 9

PRIMARY CONTACTS

Name and Responsibility	Organization and Address	Phone Number
Dr. James Elder Regional Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3536
Mr. Norrell Wallace Refuge Manager	U.S. Fish and Wildlife Service Crab Orchard National Wildlife Refuge P.O. Box J Carterville, IL 62918	618/997-3344
Dr. Dave Stallings Dr. Jim Petty Quality Control/ Quality Assurance	Columbia National Fisheries Research Laboratory U.S. Fish and Wildlife Service Route 1 Columbia, MO 65201	314/875-5399
Mr. Dick Ruelle Illinois Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service 1830 Second Avenue Rock Island, IL 61201	309/793-5800
Contracting and General Services	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3580
Mr. Richard Boice On-Scene Coordinator	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-4740
Mr. Bob Cowles Superfund Coordinator	Illinois Environmental Protection Agency 2200 Churchill Road Springfield, IL 62706	217/782-6760
Mr. Joe Stuart Illinois EPA Representative	Illinois Environmental Protection Agency 2209 West Main Marion, IL 62959	618/997-4371
Mr. Mike Carter Illinois Dept. of Conservation Representative	Regional Fish & Wildlife Manager Illinois Dept. of Conservation R.R. 4, Box 68 Benton, IL 62812	Office: 618/435-8138 Home: 618/883-5961

TABLE 9

PRIMARY CONTACTS
(Continued)

Name and Responsibility	Organization and Address	Phone Number
~~Ms. Vanessa Musgrave~~ Community Relations	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6128
Mr. Jim Ross Community Relations	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3519
Dr. Robert L. Flentge Illinois Dept. of Public Health Contact	Illinois Dept. of Public Health 525 West Jefferson Springfield, IL 62707	217/785-2439
Mr. Les Frankland Illinois Dept. of Conservation	Illinois Dept. of Conservation 424 Lincoln Tower Plaza Springfield, IL 62706	217/782-6424
Ms. Carol B. Luly Community Relations	Illinois Environmental Protection Agency 2009 Mall Street Collinsville, IL 62234	618/345-6220
Ms. Jean Hutton Office of Soliciter U.S. Department of Interior	U.S. Department of the Interior Room 4354 18th & C Streets, N.W. Washington, D.C. 20240	202/343-5301
Mr. David M. Taliaferro Attorney, U.S. EPA	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6826
Dr. Cornelius B. Murphy, Jr. O'Brien & Gere	O'Brien & Gere Engineers, Inc. P.O. Box 4873 1304 Buckley Road Syracuse, NY 13221	315/451-4700
Mr. John Hanson Beveridge & Diamond	Beveridge & Diamond, P.C. 1333 New Hampshire Ave., N.W. Washington, D.C. 20036	202/828-0285
Ms. Ellen Summer Sangamo Weston, Inc.	Sangamo Weston, Inc. P.O. Box 48400 Atlanta, GA 30362	404/449-9006

ANALYTICAL METHOD: WATER/WELLS
CLP HSL VOLATILES

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(CLP)	ppb			
1,1,1-trichloroethane	WA 85-J664	5	SEE BELOW	SEE BELOW	SEE BELOW
1,1,2,2-tetrachloroethane	WA 85-J664	5	"	"	"
1,1,2-trichloroethane	WA 85-J664	5	"	"	"
1,1-dichloroethane	WA 85-J664	5	"	"	"
1,1-dichloroethene	WA 85-J664	5	"	"	"
1,2-dichloroethane	WA 85-J664	5	"	"	"
1,2-dichloropropane	WA 85-J664	5	"	"	"
2-butanone	WA 85-J664	10	"	"	"
2-chloroethylvinyl ether	WA 85-J664	10	"	"	"
2-hexanone	WA 85-J664	10	"	"	"
4-methyl-2-pentanone	WA 85-J664	10	"	"	"
acetone	WA 85-J664	10	"	"	"
benzene	WA 85-J664	5	"	"	"
bromodichloromethane	WA 85-J664	10	"	"	"
bromoform	WA 85-J664	5	"	"	"
bromomethane	WA 85-J664	10	"	"	"
c-1,3-dichloropropene	WA 85-J664	5	"	"	"
carbon disulfide	WA 85-J664	5	"	"	"
carbon tetrachloride	WA 85-J664	5	"	"	"
chlorobenzene	WA 85-J664	5	"	"	"
chloroethane	WA 85-J664	10	"	"	"
chloroform	WA 85-J664	5	"	"	"
chloromethane	WA 85-J664	10	"	"	"
dibromochloromethane	WA 85-J664	5	"	"	"
ethyl benzene	WA 85-J664	5	"	"	"
methylene chloride	WA 85-J664	5	"	"	"
styrene	WA 85-J664	5	"	"	"
t-1,2-dichloroethene	WA 85-J664	5	"	"	"
t-1,3-dichloropropene	WA 85-J664	5	"	"	"
tetrachloroethene	WA 85-J664	5	"	"	"
toluene	WA 85-J664	5	"	"	"
total xylenes	WA 85-J664	5	"	"	"
trichloroethene	WA 85-J664	5	"	"	"
vinyl acetate	WA 85-J664	10	"	"	"
vinyl chloride	WA 85-J664	10	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E WA 85-J664 (revised 1/86).
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Recovery limits within those of Table 5.2, Exhibit E WA 85-J664 (revised 1/86).
Calibration Continuing	Each 12 hours	Minimum RF 0.003; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by samplng crew	±20% PRE waters
MS Tuning	One per day.	BFB key ions and abundance criteria must be met for all 9 ions.
Calibration Verification	Once	Five concentrations - linear range volatiles 0-500 ng.

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS

CLP HSL BASE/NEUTRALS/ACIDS (SEMI-VOLATILES)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(CLP)	ppb			
1,2,4-trichlorobenzene	WA 85-J664	10	SEE BELOW	SEE BELOW	SEE BELOW
1,2-dichlorobenzene	WA 85-J664	10	"	"	"
1,3-dichlorobenzene	WA 85-J664	10	"	"	"
1,4-dichlorobenzene	WA 85-J664	10	"	"	"
2,4,5-trichlorophenol	WA 85-J664	50	"	"	"
2,4,6-trichlorophenol	WA 85-J664	10	"	"	"
2,4-dichlorophenol	WA 85-J664	10	"	"	"
2,4-dimethylphenol	WA 85-J664	10	"	"	"
2,4-dinitrophenol	WA 85-J664	50	"	"	"
2,4-dinitrotoluene	WA 85-J664	10	"	"	"
2,6-dinitrotoluene	WA 85-J664	10	"	"	"
2-chloronaphthalene	WA 85-J664	10	"	"	"
2-chlorophenol	WA 85-J664	10	"	"	"
2-methyl-4,6-dinitrophenol	WA 85-J664	50	"	"	"
2-methylnaphtalene	WA 85-J664	10	"	"	"
2-methylphenol	WA 85-J664	10	"	"	"
2-nitroaniline	WA 85-J664	50	"	"	"
2-nitrophenol	WA 85-J664	10	"	"	"
3,3'-dichlorobenzidine	WA 85-J664	20	"	"	"
3-nitroaniline	WA 85-J664	50	"	"	"
4-bromophenyl phenyl ether	WA 85-J664	10	"	"	"
4-chloro-3-methylphenol	WA 85-J664	10	"	"	"
4-chloroaniline	WA 85-J664	10	"	"	"
4-chlorophenyl phenyl ether	WA 85-J664	10	"	"	"
4-methylphenol	WA 85-J664	10	"	"	"
4-nitroaniline	WA 85-J664	50	"	"	"
N-nitrosodi-n-propylamine	WA 85-J664	10	"	"	"
N-nitrosodiphenylamine	WA 85-J664	10	"	"	"
acenaphthalene	WA 85-J664	10	"	"	"
acenaphthene	WA 85-J664	10	"	"	"
anthracene	WA 85-J664	10	"	"	"
benzo(a)anthracene	WA 85-J664	10	"	"	"
benzo(a)pyrene	WA 85-J664	10	"	"	"
benzo(b)fluoranthene	WA 85-J664	10	"	"	"
benzo(g,h,i)perylene	WA 85-J664	10	"	"	"
benzo(k)fluoranthene	WA 85-J664	10	"	"	"
benzoic acid	WA 85-J664	50	"	"	"
benzyl alcohol	WA 85-J664	10	"	"	"
bis(2-chloroethoxy)methane	WA 85-J664	10	"	"	"
bis(2-chloroethyl) ether	WA 85-J664	10	"	"	"
bis(2-chloroisopropyl) ether	WA 85-J664	10	"	"	"
bis(2-ethylhexyl)phthalate	WA 85-J664	10	"	"	"
butyl benzyl phthalate	WA 85-J664	10	"	"	"
chrysene	WA 85-J664	10	"	"	"
di-n-butylphthalate	WA 85-J664	10	"	"	"
di-n-octyl phthalate	WA 85-J664	10	"	"	"
dibenzo(a,h)anthracene	WA 85-J664	10	"	"	"
dibenzofuran	WA 85-J664	10	"	"	"
diethyl phthalate	WA 85-J664	10	"	"	"
dimethyl phthalate	WA 85-J664	10	"	"	"
fluoranthene	WA 85-J664	10	"	"	"
fluorene	WA 85-J664	10	"	"	"
hexachlorobenzene	WA 85-J664	10	"	"	"
hexachlorobutadiene	WA 85-J664	10	"	"	"
hexachlorocyclopentadiene	WA 85-J664	10	"	"	"
hexachloroethane	WA 85-J664	10	"	"	"
indeno(1,2,3-c,d)pyrene	WA 85-J664	10	"	"	"
isophorone	WA 85-J664	10	"	"	"
naphthalene	WA 85-J664	10	"	"	"
nitrobenzene	WA 85-J664	10	"	"	"
pentachlorophenol	WA 85-J664	50	"	"	"
phenanthrene	WA 85-J664	10	"	"	"
phenol	WA 85-J664	10	"	"	"
pyrene	WA 85-J664	10	"	"	"

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS

CLP HSL BASE/NEUTRALS/ACIDS (SEMI-VOLATILES)

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E WA 85-J664.
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Recovery limits within those of Table 5.2, Exhibit E WA 85-J664.
Calibration Continuing	Each 12 hours	Minimum RF 0.05; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by sapling crew	±20% PRE waters
MS Tuning	One per day.	DFTPP key ions & abundance criteria must be met for all 13 ions.
Calibration Verification	Once	Five concentrations - linear range Base/Neutrals 0-400 ng. Acids 0-1000 ng.

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
CLP HSL NITROSAMINES (LOW LEVEL)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	*				
N-nitrosodi-n-propylamine	607	0.46	SEE BELOW	SEE BELOW	SEE BELOW
N-nitrosodimethylamine	607	0.15	"	"	"
N-nitrosodiphenylamine	607	0.81	"	"	"

* 40 CFR Part 136, October 26, 1984.
Note: If possible, lower detection limits will be attained for cleaner samples using smaller extract volumes.

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Limits within those of Table 5.2, Exhibit E WA 85-J664.
Calibration Continuing	Each 12 hours	Minimum RF 0.05; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 10-provided by sampling crew	±20% PRE waters
Calibration Verification	Once	Three concentrations - linear range nitrosamines 0 - 400 ng.

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS

CLP HSL PESTICIDES/PCBs

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(CLP)	ppb			
4,4'-DDD	WA 85-J664	0.10	SEE BELOW	SEE BELOW	SEE BELOW
4,4'-DDE	WA 85-J664	0.10	"	"	"
4,4'-DDT	WA 85-J664	0.10	"	"	"
aldrin	WA 85-J664	0.05	"	"	"
Aroclor 1016	WA 85-J664	0.5	"	"	"
Aroclor 1221	WA 85-J664	0.5	"	"	"
Aroclor 1232	WA 85-J664	0.5	"	"	"
Aroclor 1242	WA 85-J664	0.5	"	"	"
Aroclor 1248	WA 85-J664	0.5	"	"	"
Aroclor 1254	WA 85-J664	1.0	"	"	"
Aroclor 1260	WA 85-J664	1.0	"	"	"
chlordane	WA 85-J664	0.5	"	"	"
dieldrin	WA 85-J664	0.10	"	"	"
endosulfan I	WA 85-J664	0.05	"	"	"
endosulfan II	WA 85-J664	0.1	"	"	"
endosulfan sulfate	WA 85-J664	0.1	"	"	"
endrin	WA 85-J664	0.1	"	"	"
endrin ketone	WA 85-J664	0.1	"	"	"
heptachlor	WA 85-J664	0.05	"	"	"
heptachlor epoxide	WA 85-J664	0.05	"	"	"
methoxychlor	WA 85-J664	0.5	"	"	"
toxaphene	WA 85-J664	1.0	"	"	"
α-BHC	WA 85-J664	0.05	"	"	"
β-BHC	WA 85-J664	0.05	"	"	"
γ-BHC (lindane)	WA 85-J664	0.05	"	"	"
δ-BHC	WA 85-J664	0.05	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Retention Time Windows	Once per 24 hours	4,4'-DDT must have retention time greater than or equal to 12 minutes on packed column, less than 2% shift on packed and .3% for capillary column.
Evaluation Mixtures A,B, & C	Once per 72 hours.	% RSD for aldrin, endrin & dibutylchloroendate must be less than or equal to 10%.
Column Breakthrough	Once per 72 hours.	Must not exceed 20% - if greater remedial action is required.
Standard Mix	Once per 72 hours then intermittently throughout analysis	Calculated factors must not exceed 15% difference for the quantitation run nor 20% difference for confirmation run during 12-hr period. Deviation greater than or equal to 15% requires reanalysis.
Confirmation Analysis	Once per 72 hours.	Separation should be greater than or equal to 25% resolution between peaks.
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E WA 85-J664.
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Must fall within limits of Table 5.2, Exhibit E WA 85-J664.

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
PCBs (LOW LEVEL)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)*	ppt			
Aroclor 1016	608	5	SEE BELOW	SEE BELOW	SEE BELOW
Aroclor 1221	608	5	"	"	"
Aroclor 1232	608	5	"	"	"
Aroclor 1242	608	5	"	"	"
Aroclor 1248	608	5	"	"	"
Aroclor 1254	608	5	"	"	"
Aroclor 1260	608	5	"	"	"

Note: General Procedures for PCBs are included in Attachment 5, while special procedures for extraction of low level PCBs in water samples are included in Attachment 7.

* Method 608 is referenced for instrument conditions.

AUDIT	FREQUENCY	CONTROL LIMITS
Retention Time Windows	Once per 24 hours	Aroclor 1254 will be run every 10 samples, less than 2% shift on packed and .3% for capillary column.
Aroclor 1254	Once per 72 hours.	% RSD for Aroclor 1254 & dibutylchloroendate (or equivalent) must be less than or equal to 10%.
Standard Aroclor Mix	Once per 72 hours then intermittently throughout analysis	Calculated factors must not exceed 15% difference for the quantitation run nor 20% difference for confirmation run during 12-hr period. Deviation greater than or equal to 15% requires reanalysis.
Confirmation Analysis	Once per 72 hours.	Separation should be greater than or equal to 25% resolution between peaks.
Reagent Blank	1 per case or 1 in 20 of similar concentration/matrix.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within 27-154.
MS/MSD	1 in 5 samples of similar concentration/matrix.	Must fall within limits of ±25%.
Dual Column Analysis	Only positive analysis	Confirmation on mixed phase 1.5% SP 2250 and 1.95% SP2401.

Lock range for 1254

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
AA METALS

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb			
aluminum	WA85-J838,9	200	SEE BELOW	SEE BELOW	SEE BELOW
antimony	WA85-J838,9	60	"	"	"
arsenic	WA85-J838,9	10	"	"	"
barium	WA85-J838,9	200	"	"	"
beryllium	WA85-J838,9	5	"	"	"
cadmium	WA85-J838,9	5	"	"	"
calcium	WA85-J838,9	5000	"	"	"
chromium	WA85-J838,9	10	"	"	"
cobalt	WA85-J838,9	50	"	"	"
copper	WA85-J838,9	25	"	"	"
iron	WA85-J838,9	100	"	"	"
lead	WA85-J838,9	5	"	"	"
magnesium	WA85-J838,9	1000	"	"	"
manganese	WA85-J838,9	15	"	"	"
mercury (cold vapor)	WA85-J838,9	0.2	"	"	"
nickel	WA85-J838,9	40	"	"	"
potassium	WA85-J838,9	5000	"	"	"
selenium	WA85-J838,9	5	"	"	"
silver	WA85-J838,9	10	"	"	"
sodium	WA85-J838,9	5000	"	"	"
thallium	WA85-J838,9	10	"	"	"
vanadium	WA85-J838,9	50	"	"	"
zinc	WA85-J838,9	20	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	Calibrated daily and each time instrument is set up; verify at at a frequency of 10% or every 2 hr, whichever is greater.	Within ±10% of true value for all except tin and mecury (±20% of true value).
Calibration Blank	During calibration at a frequency of 10% during run and at end of run.	No more than CRDL.
Preparation Blank	1 per batch of samples digested or 1 in 20 whichever is greater	No more than CRDL.
Spiked Sample Analysis	1 per group of similar concentration and matrix, 1 per case of samples, or 1 in 20, whichever is greater.	Within ±25% recovery
Duplicate Sample Analysis	Same as spiked sample analysis.	±20% RPD for values 5X CRDL or more ±CRDL for samples less than 5X CRDL
Lab Control Sample (aqueous)	1 for each procedure for each case of samples received; 1 in 20 or 1 per batch digested, whichever is greater.	Within 80-120% recovery
Spike Sample	each analysis	In accordance with limits shown in Section 7, Exhibit E, SOW no. 784 (July ~~1984~~ 1985)

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pages - 7, 9, 10, 20, 21

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS

AA METALS

Dissolved Metals: Those constituents (metals) which will pass through a 0.45µ membrane filter.

Field Filtration Protocol:

An aliquot of sample will be passed through a 0.45µ membrane filter by one of the following methods:

1) Plastic syringe equipped with a filter holder (Swinnex Filter Holder).
2) Hand vacuum pump and a 500 ml side arm, glass filtration flask.
3) Bench top (electric) filtration system.

•Standards and samples will be matrix-matched to the concentration of the mineral acid.

•Calibration curves, continuing calibration and corrective measures records will be documented.

•One medium range internal synthetic standard will be analyzed to verify calibration and will be within ±10% of true value

•Furnace work will require ~~duplicate~~ spike analysis of each sample to verify recovery of spiked material. If recoveries are within ±10%, methods of addition will not be required. If outside this criterion, methods of standard addition will be required.

•For chromium analysis, a nitrous oxide flame will be used.

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
NIPDWR METALS (40 CFR 141)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	*	ppb	SEE BELOW	SEE BELOW	SEE BE
Arsenic	EPA 206.2	5	"	"	"
Barium	EPA 208.1	100	"	"	"
Cadmium	EPA 213.2	1	"	"	"
Chromium	EPA 218.2	1	"	"	"
Lead	EPA 239.2	~~5~~ 1.2	"	"	"
Mercury	EPA 245.1	~~1~~ 1.2	"	"	"
Selenium	EPA 270.2	5	"	"	"
Silver	EPA 272.1	10	"	"	"

* Methods reference: AA by flame or furnace.

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	Calibrated daily and each time instrument is set up; verify at at a frequency of 10% or every 2 hr, whichever is greater.	Within ±10% of true value for all except tin and mecury (±20% of true value).
Calibration Blank	During calibration at a frequency of 10% during run and at end of run.	No more than CRDL.
Preparation Blank	1 per batch of samples digested or 1 in 20 whichever is greater	No more than CRDL.
Spiked Sample Analysis	1 per group of similar concentration and matrix, 1 per case of samples, or 1 in 20, whichever is greater.	Within ±25% recovery
Duplicate Sample Analysis	Same as spiked sample analysis.	±20% RPD for values 5X CRDL or more ±CRDL for samples less than 5X CRDL
Lab Control Sample (aqueous)	1 for each procedure for each case of samples received; 1 in 20 or 1 per batch digested, whichever is greater.	Within 80-120% recovery
Spike Sample	each analysis	In accordance with limits shown in Section 7, Exhibit E, SOW no. 784 (July 1984)

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
SPECIAL METALS

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	no method #	ppb			
arsenic	206.2	5	SEE BELOW	SEE BELOW	SEE BELOW
cadmium	213.2	1	"	"	"
chromium	218.2	1	"	"	"
copper	220.2	1	"	"	"
lead	239.2	~~0.2~~ /	"	"	"
magnesium	242.1	1000	"	"	"
mercury	245.2	0.2	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	Calibrated daily and each time instrument is set up; verify at at a frequency of 10% or every 2 hr, whichever is greater.	Within ±10% of true value for all except tin and mecury (±20% of true value).
Calibration Blank	During calibration at a frequency of 10% during run and at end of run.	No more than CRDL.
Preparation Blank	1 per batch of samples digested or 1 in 20 whichever is greater	No more than CRDL.
Spiked Sample Analysis	1 per group of similar concentration and matrix, 1 per case of samples, or 1 in 20, whichever is greater.	Within ±25% recovery
Duplicate Sample Analysis	Same as spiked sample analysis.	±20% RPD for values 5X CRDL or more ±CRDL for samples less than 5X CRDL
Lab Control Sample (aqueous)	1 for each procedure for each case of samples received; 1 in 20 or 1 per batch digested, whichever is greater.	Within 80-120% recovery
Spike Sample	each analysis	In accordance with limits shown in Section 7, Exhibit E, SOW no. 784 (July 1984)

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
WET CHEMISTRY

CHEMICAL COMPOUND	METHOD *	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb			
ammonia nitrogen	350.1	10	SEE BELOW	SEE BELOW	SEE BELOW
cyanide 335.3	~~335.1~~ — ?	10	"	"	"
fluoride	340.2	100	"	"	"
nitrate + nitrite as N	353.1	10	"	"	"
nitrate nitrogen	352.1	10	"	"	"
percent solids	160.3	0.1 %	"	"	"
pH	150.1	0.1 std units	"	"	"
total phosphorus	365.4	10	"	"	"

* Methods Reference: EPA-600/4-79-020 "Methods for Chemical Analysis of Water and Waste Waters"

** Standard Methods for the Evaluation of Water and Wastewater. 16th Ed. 1985.

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	calibrated daily and each time instrument is set up; verify at a frequency of 10% or every 2 whichever is greater.	Within ±10% of true value.
Calibration Blank	during calibration, at a frequency of 10% during run, and at end of run.	No more than CRDL
Preparation Blank	1 per batch of samples or 1 in 20, whichever is greater.	No more than CRDL
Duplicate Sample Analysis	1 per case of samples or 1 in 20, whichever is greater.	±20% RPD for values 5X CRDL or more; ±CRDL for samples less than 5X CRDL.
Spiked Sample Analysis	1 per group of similar concentration, 1 per case of samples, or 1 in 20; 1 at end of run for nitrate and nitrite.	within ±25% recovery

See last page for abbreviations.

ANALYTICAL METHOD: WATER/WELLS
EXPLOSIVES

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb			
1,3 DNB	SEE NOTE 6	0.5			
1,3,5 TNB	SEE NOTE 6	0.5			
2,4 DNT	SEE NOTE 6	0.5			
2,4,6 TNT	SEE NOTE 6	0.7			
2,6 DNT	SEE NOTE 6	0.5			
HMX	SEE NOTE 6	2.0			
NB	SEE NOTE 6	0.4			
RDX	SEE NOTE 6	1.2			
tetryl	SEE NOTE 6	0.6			

NOTE 6 USATHAMA Method 2C Cyclotrimethylenetrinitriteamine (RDX) samples, 12/8/80.
See abbreviations on p.16.

ANALYTICAL METHOD: SOIL/SEDIMENT
CLP HSL VOLATILES

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
1,1,1-trichloroethane	WA 85-J664	5	SEE BELOW	SEE BELOW	SEE BELOW
1,1,2,2-tetrachloroethane	WA 85-J664	5	"	"	"
1,1,2-trichloroethane	WA 85-J664	5	"	"	"
1,1-dichloroethane	WA 85-J664	5	"	"	"
1,1-dichloroethene	WA 85-J664	5	"	"	"
1,2-dichloroethane	WA 85-J664	5	"	"	"
1,2-dichloropropane	WA 85-J664	5	"	"	"
2-butanone	WA 85-J664	10	"	"	"
2-chloroethylvinyl ether	WA 85-J664	10	"	"	"
2-hexanone	WA 85-J664	10	"	"	"
4-methyl-2-pentanone	WA 85-J664	10	"	"	"
acetone	WA 85-J664	10	"	"	"
benzene	WA 85-J664	5	"	"	"
bromodichloromethane	WA 85-J664	10	"	"	"
bromoform	WA 85-J664	5	"	"	"
bromomethane	WA 85-J664	10	"	"	"
c-1,3-dichloropropene	WA 85-J664	5	"	"	"
carbon disulfide	WA 85-J664	5	"	"	"
carbon tetrachloride	WA 85-J664	5	"	"	"
chlorobenzene	WA 85-J664	5	"	"	"
chloroethane	WA 85-J664	10	"	"	"
chloroform	WA 85-J664	5	"	"	"
chloromethane	WA 85-J664	10	"	"	"
dibromochloromethane	WA 85-J664	5	"	"	"
ethyl benzene	WA 85-J664	5	"	"	"
methylene chloride	WA 85-J664	5	"	"	"
styrene	WA 85-J664	5	"	"	"
t-1,2-dichloroethene	WA 85-J664	5	"	"	"
t-1,3-dichloropropene	WA 85-J664	5	"	"	"
tetrachloroethene	WA 85-J664	5	"	"	"
toluene	WA 85-J664	5	"	"	"
total xylenes	WA 85-J664	5	"	"	"
trichloroethene	WA 85-J664	5	"	"	"
vinyl acetate	WA 85-J664	10	"	"	"
vinyl chloride	WA 85-J664	10	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E WA 85-J664 (revised 1/86).
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Recovery limits within those of Table 5.2, Exhibit E WA 85-J664 (revised 1/86).
Calibration Continuing	Each 12 hours	Minimum RF 0.003; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by sampling crew	±50% PRE soils
MS Tuning	One per day.	BFB key ions and abundance criteria must be met for all 9 ions.
Calibration Verification	Once	Five concentrations - linear range volatiles 0-500 ng.

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT

CLP HSL BASE/NEUTRALS/ACIDS (SEMI-VOLATILES)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(CLP)	ppb			
1,2,4-trichlorobenzene	WA 85-J664	330	SEE BELOW	SEE BELOW	SEE BELOW
1,2-dichlorobenzene	WA 85-J664	330	"	"	"
1,3-dichlorobenzene	WA 85-J664	330	"	"	"
1,4-dichlorobenzene	WA 85-J664	330	"	"	"
2,4,5-trichlorophenol	WA 85-J664	1600	"	"	"
2,4,6-trichlorophenol	WA 85-J664	330	"	"	"
2,4-dichlorophenol	WA 85-J664	330	"	"	"
2,4-dimethylphenol	WA 85-J664	330	"	"	"
2,4-dinitrophenol	WA 85-J664	1600	"	"	"
2,4-dinitrotoluene	WA 85-J664	330	"	"	"
2,6-dinitrotoluene	WA 85-J664	330	"	"	"
2-chloronaphthalene	WA 85-J664	330	"	"	"
2-chlorophenol	WA 85-J664	330	"	"	"
2-methyl-4,6-dinitrophenol	WA 85-J664	1600	"	"	"
2-methylnaphtalene	WA 85-J664	330	"	"	"
2-nitroaniline	WA 85-J664	1600	"	"	"
2-nitrophenol	WA 85-J664	330	"	"	"
3,3'-dichlorobenzidine	WA 85-J664	660	"	"	"
3-nitroaniline	WA 85-J664	1600	"	"	"
4-bromophenyl phenyl ether	WA 85-J664	330	"	"	"
4-chloro-3-methylphenol	WA 85-J664	330	"	"	"
4-chloroaniline	WA 85-J664	330	"	"	"
4-chlorophenyl phenyl ether	WA 85-J664	330	"	"	"
4-nitroaniline	WA 85-J664	1600	"	"	"
N-nitrosodi-n-propylamine	WA 85-J664	330	"	"	"
N-nitrosodiphenylamine	WA 85-J664	330	"	"	"
acenaphthalene	WA 85-J664	330	"	"	"
acenaphthene	WA 85-J664	330	"	"	"
anthracene	WA 85-J664	330	"	"	"
benzo(a)anthracene	WA 85-J664	330	"	"	"
benzo(a)pyrene	WA 85-J664	330	"	"	"
benzo(b)fluoranthene	WA 85-J664	330	"	"	"
benzo(g,h,i)perylene	WA 85-J664	330	"	"	"
benzo(k)fluoranthene	WA 85-J664	330	"	"	"
benzoic acid	WA 85-J664	1600	"	"	"
benzyl alcohol	WA 85-J664	330	"	"	"
bis(2-chloroethoxy)methane	WA 85-J664	330	"	"	"
bis(2-chloroethyl) ether	WA 85-J664	330	"	"	"
bis(2-chloroisopropyl) ether	WA 85-J664	330	"	"	"
bis(2-ethylhexyl)phthalate	WA 85-J664	330	"	"	"
butyl benzyl phthalate	WA 85-J664	330	"	"	"
chrysene	WA 85-J664	330	"	"	"
di-n-butylphthalate	WA 85-J664	330	"	"	"
di-n-octyl phthalate	WA 85-J664	330	"	"	"
dibenzo(a,h)anthracene	WA 85-J664	330	"	"	"
dibenzofuran	WA 85-J664	330	"	"	"
diethyl phthalate	WA 85-J664	330	"	"	"
dimethyl phthalate	WA 85-J664	330	"	"	"
fluoranthene	WA 85-J664	330	"	"	"
fluorene	WA 85-J664	330	"	"	"
hexachlorobenzene	WA 85-J664	330	"	"	"
hexachlorobutadiene	WA 85-J664	330	"	"	"
hexachlorocyclopentadiene	WA 85-J664	330	"	"	"
hexachloroethane	WA 85-J664	330	"	"	"
indeno(1,2,3-c,d)pyrene	WA 85-J664	330	"	"	"
isophorone	WA 85-J664	330	"	"	"
naphthalene	WA 85-J664	330	"	"	"
nitrobenzene	WA 85-J664	330	"	"	"
pentachlorophenol	WA 85-J664	1600	"	"	"
phenanthrene	WA 85-J664	330	"	"	"
phenol	WA 85-J664	330	"	"	"
pyrene	WA 85-J664	330	"	"	"

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT

CLP HSL BASE/NEUTRALS/ACIDS (SEMI-VOLATILES)

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E WA 85-J664.
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Recovery limits within those of Table 5.2, Exhibit E WA 85-J664.
Calibration Continuing	Each 12 hours	Minimum RF 0.05; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by smplng crew	±50% PRE soils
MS Tuning	One per day.	DFTPP key ions & abundance criteria must be met for all 13 ions.
Calibration Verification	Once	Five concentrations - linear range Base/Neutrals 0-400 ng. Acids 0-1000 ng.

See last page for abbreviations.

Table 10, Page 16 of 25
ANALYTICAL METHOD: SOIL/SEDIMENT
CLP HSL NITROSAMINES (LOW LEVEL)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	*	ppb			
N-nitrosodi-n-propylamine	607	330	SEE BELOW	SEE BELOW	SEE BELOW
N-nitrosodimethylamine	607	330	"	"	"
N-nitrosodiphenylamine	607	330	"	"	"

* 40 CFR Part 136, October 26, 1984.

Notes: If possible, lower detection limits will be obtained for cleaner samples using lower extraction volumes.

Extraction procedures will follow U.S. EPA SW-846 - Method 8250. Quantification and quantitation by Method 607.

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Limits within those of Table 5.2, Exhibit E WA 85-J664.
Calibration Continuing	Each 12 hours	Minimum RF 0.05; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by smpling crew	±50% PRE soils
Calibration Verification	Once	Three concentrations - linear range nitrosamines 0 - 400 ng.

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
CLP HSL PESTICIDES/PCBs

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	AUDIT	FREQUENCY	CONTROL LIMITS
	(CLP)	ppb					
4,4'-DDD	WA 85-J664	16	SEE BELOW	SEE BELOW	SEE BELOW		
4,4'-DDE	WA 85-J664	16	"	"	"		
4,4'-DDT	WA 85-J664	16	"	"	"		
aldrin	WA 85-J664	8	"	"	"		
Aroclor 1016	WA 85-J664	80	"	"	"		
Aroclor 1221	WA 85-J664	80	"	"	"		
Aroclor 1232	WA 85-J664	80	"	"	"		
Aroclor 1242	WA 85-J664	60	"	"	"		
Aroclor 1248	WA 85-J664	80	"	"	"		
Aroclor 1254	WA 85-J664	160	"	"	"		
Aroclor 1260	WA 85-J664	160	"	"	"		
chlordane	WA 85-J664	80	"	"	"		
dieldrin	WA 85-J664	16	"	"	"		
endosulfan I	WA 85-J664	8.0	"	"	"		
endosulfan II	WA 85-J664	16	"	"	"		
endosulfan sulfate	WA 85-J664	16	"	"	"		
endrin	WA 85-J664	16	"	"	"		
endrin ketone	WA 85-J664	16	"	"	"		
heptachlor	WA 85-J664	8.0	"	"	"		
heptachlor epoxide	WA 85-J664	8.0	"	"	"		
methoxychlor	WA 85-J664	80	"	"	"		
toxaphene	WA 85-J664	160	"	"	"		
α-BHC	WA 85-J664	8.0	"	"	"		
β-BHC	WA 85-J664	8.0	"	"	"		
γ-BHC (lindane)	WA 85-J664	8.0	"	"	"		
δ-BHC	WA 85-J664	8.0	"	"	"		

AUDIT	FREQUENCY	CONTROL LIMITS
Retention Time Windows	Once per 24 hours	4,4'-DDT must have retention time greater than or equal to 12 minutes on packed column, less than 2% shift on packed and .3% for capillary column.
Evaluation Mixtures A, B, & C	Once per 72 hours.	% RSD for aldrin, endrin & dibutylchloroendate must be less than or equal to 10%.
Column Breakthrough	Once per 72 hours.	Must not exceed 20% - if greater remedial action is required.
Standard Mix	Once per 72 hours then intermittently throughout analysis	Calculated factors must not exceed 15% difference for the quantitation run nor 20% difference for confirmation run during 12-hr period. Deviation greater than or equal to 15% requires reanalysis.
Confirmation Analysis	Once per 72 hours.	Separation should be greater than or equal to 25% resolution between peaks.
Reagent Blank	1 per case or 5% of sample shipment.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E WA 85-J664.
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Must fall within limits of Table 5.2, Exhibit E WA 85-J664.

* A detection level of 1.0 ppm will be used for Area 9 Building Complex.

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
PCBs, GENERAL

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)*	ppb			
Aroclor 1016	608	500	(See Attachment 6.)		
Aroclor 1221	608	500	"	"	"
Aroclor 1232	608	500	"	"	"
Aroclor 1242	608	500	"	"	"
Aroclor 1248	608	500	"	"	"
Aroclor 1254	608	500	"	"	"
Aroclor 1260	608	500	"	"	"

* Note: General procedures for extraction of PCBs are included in Attachment 5. #6 ~~A~~ extraction is not included!

AUDIT	FREQUENCY	CONTROL LIMITS
Retention Time Windows	Once per 24 hours	Aroclor 1254 will be run every 10 samples, less than 2% shift on packed and .3% for capillary column.
Aroclor 1254	Once per 72 hours.	% RSD for Aroclor 1254 & dibutylchloroendate (or equivalent) must be less than or equal to 10%.
Standard Aroclor Mix	Once per 72 hours then intermittently throughout analysis	Calculated factors must not exceed 15% difference for the quantitation run nor 20% difference for confirmation run during 12-hr period. Deviation greater than or equal to 15% requires reanalysis.
Confirmation Analysis	Once per 72 hours.	Separation should be greater than or equal to 25% resolution between peaks.
Reagent Blank	1 per case or 1 in 20 of similar concentration/matrix.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within 27-154.
MS/MSD	1 in 5 samples of similar concentration/matrix.	Must fall within limits of ±25%.
Dual Column Analysis	Only positive analysis	Confirmation on mixed phase 1.5% SP 2250 and 1.95% SP2401.

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
PCBs (SEMI-LOW LEVEL)

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)*	ppb			
Aroclor 1016	608	40	(See Attachment 6.)		
Aroclor 1221	608	40	"	"	"
Aroclor 1232	608	40	"	"	"
Aroclor 1242	608	40	"	"	"
Aroclor 1248	608	40	"	"	"
Aroclor 1254	608	40	"	"	"
Aroclor 1260	608	40	"	"	"

Note: General procedures for extraction of PCBs are included in Attachment 5, while special procedures for low level PCBs extraction are included in Attachment 6.

AUDIT	FREQUENCY	CONTROL LIMITS
Retention Time Windows	Once per 24 hours	Aroclor 1254 will be run every 10 samples, less than 2% shift on packed and .3% for capillary column.
Aroclor 1254	Once per 72 hours.	% RSD for Aroclor 1254 & dibutylchloroendate (or equivalent) must be less than or equal to 10%.
Standard Aroclor Mix	Once per 72 hours then intermittently throughout analysis	Calculated factors must not exceed 15% difference for the quantitation run nor 20% difference for confirmation run during 12-hr period. Deviation greater than or equal to 15% requires reanalysis.
Confirmation Analysis	Once per 72 hours.	Separation should be greater than or equal to 25% resolution between peaks.
Reagent Blank	1 per case or 1 in 20 of similar concentration/matrix.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within 27-154.
MS/MSD	1 in 5 samples of similar concentration/matrix.	Must fall within limits of ±25%.
Dual Column Analysis	Only positive analysis	Confirmation on mixed phase 1.5% SP 2250 and 1.95% SP2401.

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
AA METALS

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	*	ppb			
aluminum	WA85-J838,9	10000	SEE BELOW	SEE BELOW	SEE BELOW
antimony	WA85-J838,9	20000	"	"	"
arsenic	WA85-J838,9	100	"	"	"
barium	WA85-J838,9	10000	"	"	"
beryllium	WA85-J838,9	500	"	"	"
cadmium	WA85-J838,9	500	"	"	"
calcium	WA85-J838,9	1000	"	"	"
chromium	WA85-J838,9	5000	"	"	"
cobalt	WA85-J838,9	5000	"	"	"
copper	WA85-J838,9	2000	"	"	"
iron	WA85-J838,9	3000	"	"	"
lead	WA85-J838,9	10000	"	"	"
magnesium	WA85-J838,9	5000	"	"	"
manganese	WA85-J838,9	1000	"	"	"
mercury (cold vapor)	WA85-J838,9	200	"	"	"
nickel	WA85-J838,9	4000	"	"	"
potassium	WA85-J838,9	1000	"	"	"
selenium	WA85-J838,9	200	"	"	"
silver	WA85-J838,9	1000	"	"	"
sodium	WA85-J838,9	1000	"	"	"
vanadium	WA85-J838,9	20000	"	"	"
zinc	WA85-J838,9	500	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	Calibrated daily and each time instrument is set up; verify at at a frequency of 10% or every 2 hr, whichever is greater.	Within ±10% of true value for all except tin and mecury (±20% of true value).
Calibration Blank	During calibration at a frequency of 10% during run and at end of run.	No more than CRDL.
Preparation Blank	1 per batch of samples digested or 1 in 20 whichever is greater	No more than CRDL.
Spiked Sample Analysis	1 per group of similar concentration and matrix, 1 per case of samples, or 1 in 20, whichever is greater.	Within ±25% recovery
Duplicate Sample Analysis	Same as spiked sample analysis.	±50% RPD for values 5X CRDL or more ±CRDL for samples less than 5X CRDL
Lab Control Sample (soils)	once a month for each of the procedures (applied) to solid sample analysis.	Within limits established by EPA.
Spike Sample	each analysis	In accordance with limits shown in Section 7, Exhibit E, SOW no. 784 (July 1984)

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
SPECIAL METALS

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb			
arsenic	206.2	100	SEE BELOW	SEE BELOW	SEE BELOW
cadmium	213.1	1000	"	"	"
chromium	218.1	5000	"	"	"
copper	220.1	2000	"	"	"
lead	239.1	8000	"	"	"
magnesium	242.1	5000	"	"	"
mercury	245.1	20	"	"	"

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	Calibrated daily and each time instrument is set up; verify at at a frequency of 10% or every 2 hr, whichever is greater.	Within ±10% of true value for all except tin and mecury (±20% of true value).
Calibration Blank	During calibration at a frequency of 10% during run and at end of run.	No more than CRDL.
Preparation Blank	1 per batch of samples digested or 1 in 20 whichever is greater	No more than CRDL.
Spiked Sample Analysis	1 per group of similar concentration and matrix, 1 per case of samples, or 1 in 20, whichever is greater.	Within ±25% recovery
Duplicate Sample Analysis	Same as spiked sample analysis.	±50% RPD for values 5X CRDL or more ±CRDL for samples less than 5X CRDL
Lab Control Sample (soils)	once a month for each of the procedures (applied) to solid sample analysis.	Within limits established by EPA.
Spike Sample	each analysis	In accordance with limits shown in Section 7, Exhibit E, SOW no. 784 (July 1984)

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
WET CHEMISTRY

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
	*	ppb			
cyanide	335.3 335.2	2000	SEE NOTE 1	"	"
percent solids	160.3	0.1 %		"	"
pH	150.1	1 S.U.	SEE NOTE 1	"	"
total phosphorus	365.4	1000	SEE NOTE 2	"	"

* - Method Reference: EPA-600/4-79-020 "Methods for Chemical Analysis of Water and Waste"

NOTE 1 SLUDGE/SOIL/SEDIMENT Aliquot are extracted with distilled deionized water for 24 hours and the supernant is analized by the referenced aqueous procedure

NOTE 2 A portion of the SLUDGE/SOIL/SEDIMENT is subjected to the block digestion procedure the resultant digestate is analyzed by the referenced procedure.

~~NOTE 3 A SLUDGE/SOIL/SEDIMENT sample is extracted with ethyl acetate and the extract is pyrolized for TOX.~~

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	calibrated daily and each time instrument is set up; verify at a frequency of 10% or every 2 whichever is greater.	Within ±10% of true value.
Calibration Blank	during calibration, at a frequency of 10% during run, and at end of run.	No more than CRDL
Preparation Blank	1 per batch of samples or 1 in 20, whichever is greater.	No more than CRDL
Duplicate Sample Analysis	1 per case of samples or 1 in 20, whichever is greater.	±50% RPD for values 5X CRDL or more; ±CRDL for samples less than 5X CRDL.
Spiked Sample Analysis	1 per group of similar concentration, 1 per case of samples, or 1 in 20; 1 at end of run for nitrate and nitrite.	within ±40% recovery

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
DIOXINS/FURANS

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
		ppt			
tetra-CDD	SEE NOTE 9	20			
tetra-CDF	SEE NOTE 9	20			
penta-CDD	SEE NOTE 9	20			
penta-CDF	SEE NOTE 9	20			
hexa-CDD	SEE NOTE 9	20			
hexa-CDF	SEE NOTE 9	20			
hepta-CDD	SEE NOTE 9	200			
hepta-CDF	SEE NOTE 9	200			
octa-CDD	SEE NOTE 9	200			
octa-CDf	SEE NOTE 9	200			

NOTE 9 Determination od Parts-per-Trillion levels of polychlorinated Dibenzolfuran and dioxins in environmental samples, Smith L.M., Johnson J.C., Analytic Chemistry 1984, 56, 1830-1842, September 1984.

See last page for abbreviations.

ANALYTICAL METHOD: SOIL/SEDIMENT
EXPLOSIVES

CHEMICAL COMPOUND	METHOD	DETECTION LIMIT	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb			
1,3 DNB	SEE NOTE 6	500			
1,3,5 TNB	SEE NOTE 6	500			
2,4 DNT	SEE NOTE 6	500			
2,4,6 TNT	SEE NOTE 6	500			
2,6 DNT	SEE NOTE 6	500			
HMX	SEE NOTE 6	500			
NB	SEE NOTE 6	500			
RDX	SEE NOTE 6	500			
tetryl	SEE NOTE 6	500			

NOTE 6 USATHAMA Method 2C Cyclotrimethylenetrinitriteamine (RDX) in soil and sediment samples, 12/8/80.

See last page for abbreviations.

ABBREVIATIONS USED

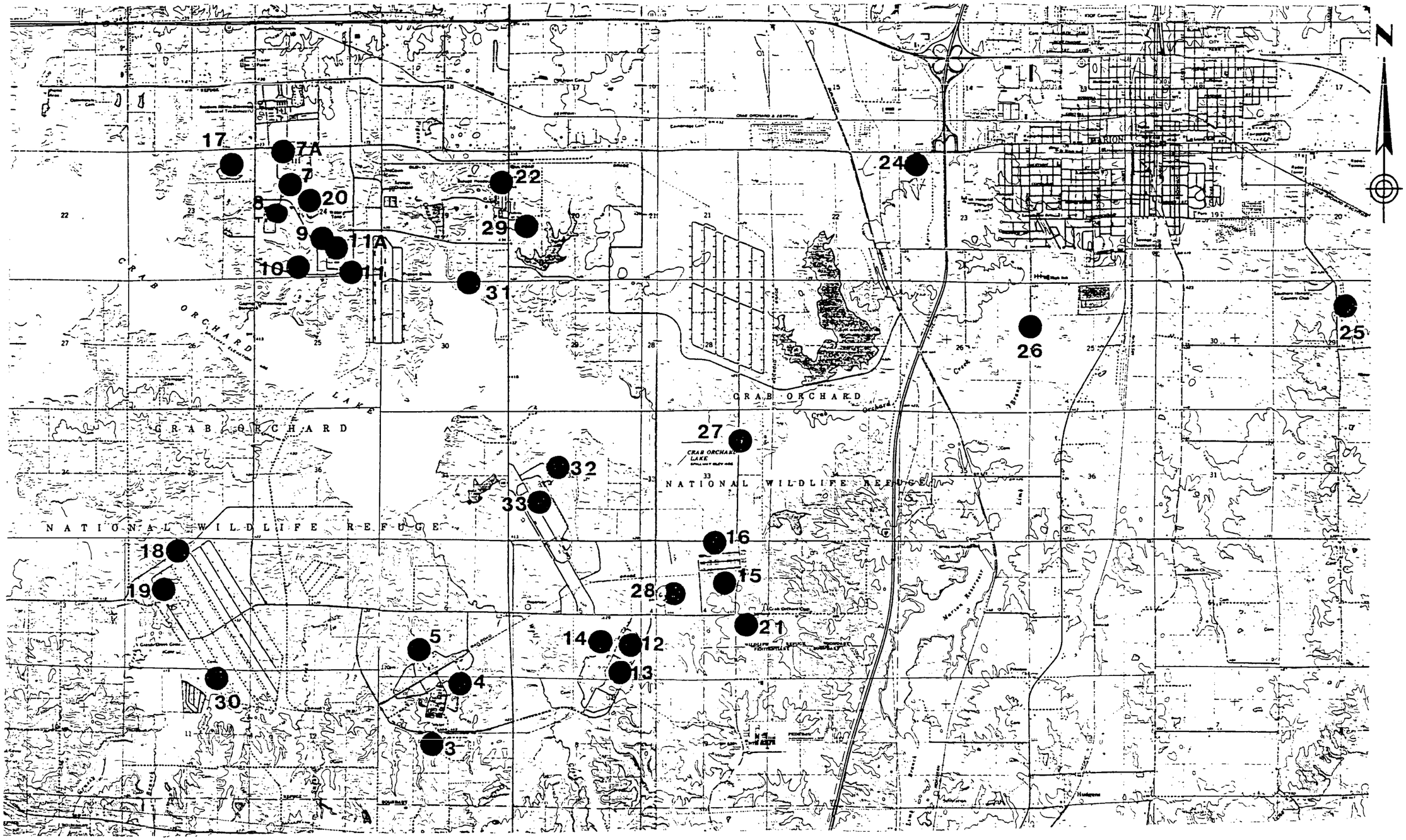
CRDL	-	CONTRACT REQUIRED DETECTION LIMITS
RF	-	RESPONSE FACTOR
PRE	-	PERCENT RELATIVE ERROR
MS/MSD	-	MATRIX SPIKE/MATRIX SPIKE DUPLICATE
RPD	-	RELATIVE PERCENT DIFFERENCE
RSD	-	RELATIVE STANDARD DEVIATION
ppb	-	PARTS PER BILLION
ppt	-	PARTS PER TRILLION
AA	-	ATOMIC ABSORPTION
GC	-	GAS CHROMATOGRAPH
GC/MS	-	GAS CHROMATOGRAPH/MASS SPECTROMETER
CLP	-	CONTRACT LABORATORY PROTOCOL
DFTPP	-	DECAFLUOROTRIPHENYL PHOSPHINE

See last page for abbreviations.

Figures

O'BRIEN & GERE

FIGURE 1



CRAB ORCHARD NATIONAL WILDLIFE REFUGE

LOCATIONS OF SAMPLING SITES

Scale: 1" – 4000'

O'BRIEN & GERE

CRAB ORCHARD NATIONAL WILDLIFE REFUGE
REVISED PROJECT SCHEDULE
Based on Phase II beginning:
a. November 1, 1986
b. March 1, 1987

Revised Oct. 14, 1986

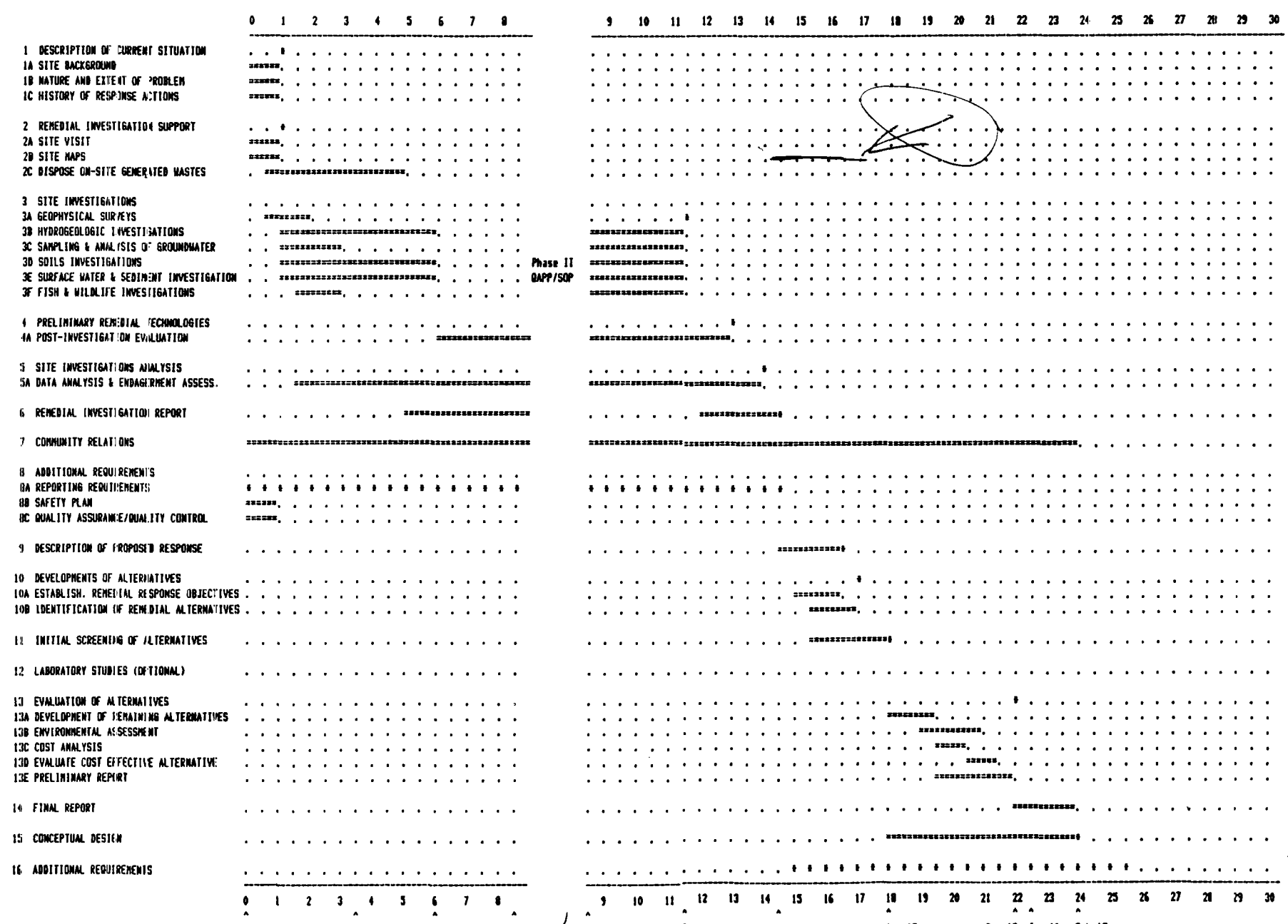


FIGURE 2

FIGURE 3

PROJECT ORGANIZATION

REMEDIAL INVESTIGATION/FEASIBILITY STUDY
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

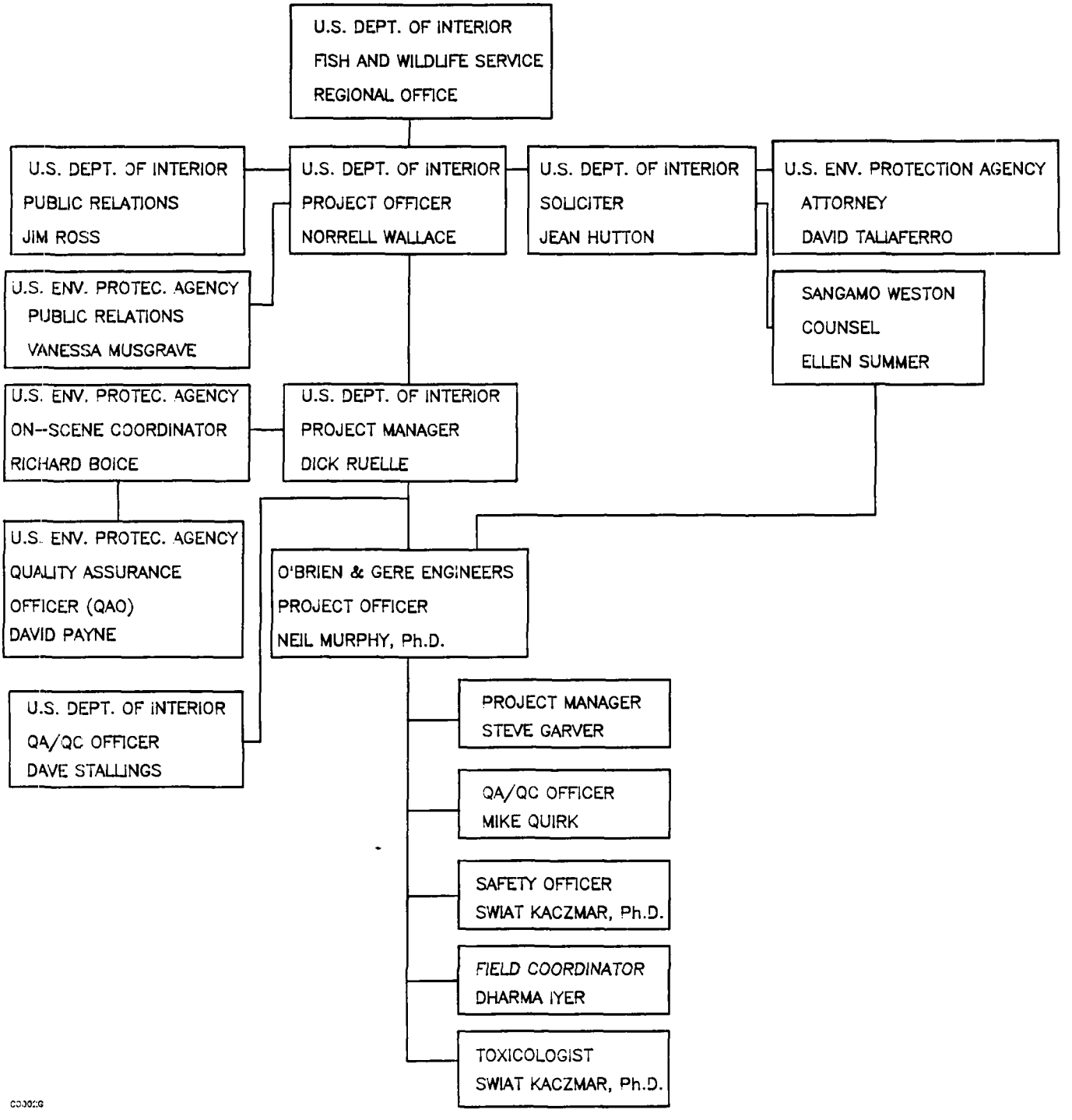
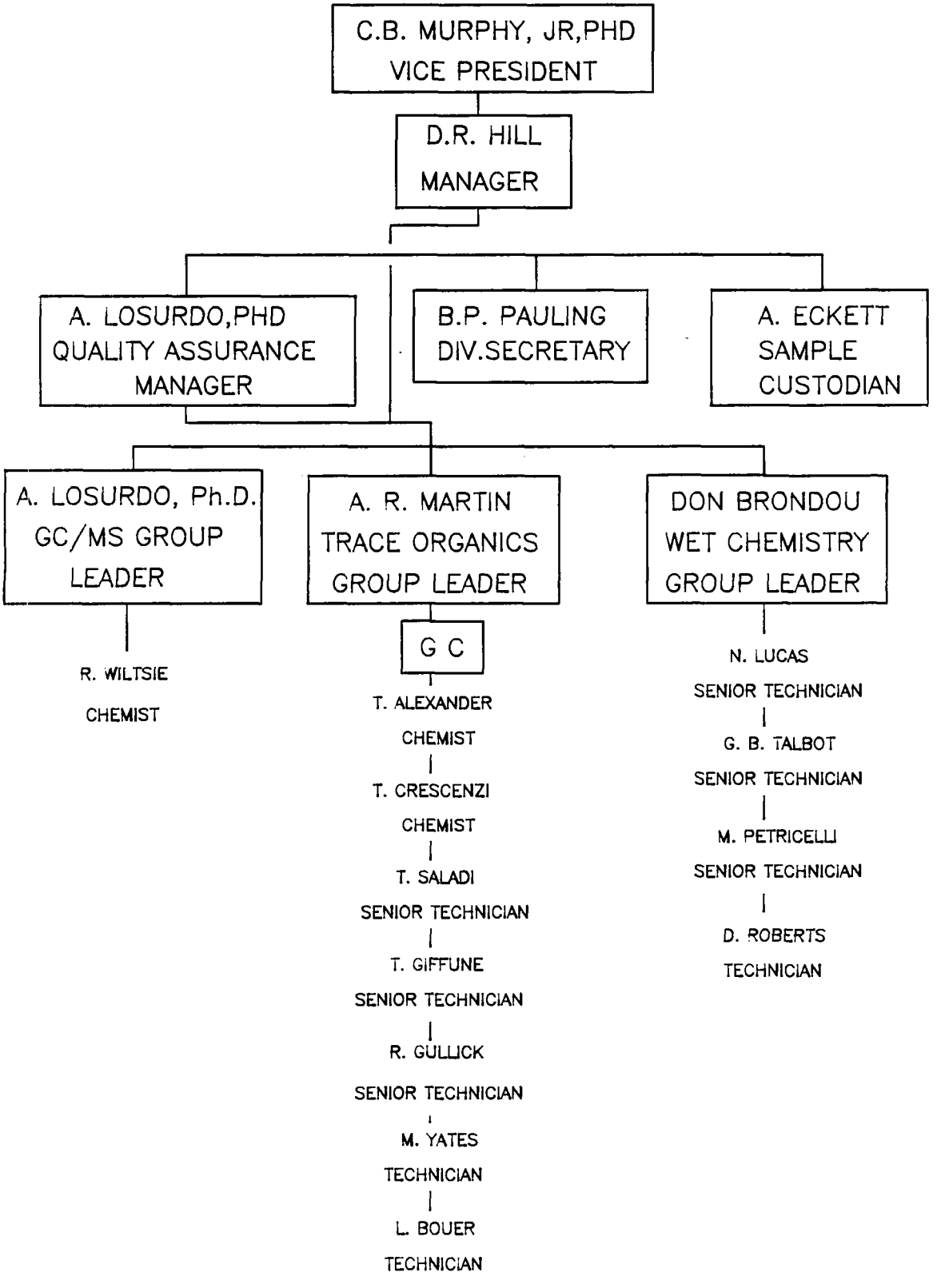


FIGURE 4

LABORATORY ORGANIZATION CHART



OBG
LABORATORIES, INC.

FIELD
CHAIN OF CUSTODY RECORD

SURVEY

SAMPLERS: *(Signature)*

STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ. NO.	NO. OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp.	Grab.				

Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by Mobile Laboratory for field analysis: *(Signature)*	Date/Time
Dispatched by: *(Signature)*	Date/Time / Received for Laboratory by:	Date/Time
Method of Shipment:		

OBG Laboratories, Inc.
Box 4873 / 1304 Buckley Road / Syracuse, New York 13221 / (315) 457-1494
Oakdale Medical Building / 700 Harry L. Drive / Johnson City, New York 13790

Attachments

O'BRIEN & GERE

ATTACHMENT 1

ANALYTICAL PROTOCOLS FOR EXPLOSIVES IN SOIL

METHOD NO.: 8H

DATE: 4-21-83

EXPLOSIVES IN SOIL BY HPLC

1. APPLICATION: Determination of the following nitro-compounds in soil.

HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
RDX	Hexahydro-1,3,5-trinitro-s-triazine
NB	Nitrobenzene
1,3-DNB	1,3-Dinitrobenzene
1,3,5-TNB	1,3,5-Trinitrobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
2,4,6-TNT	2,4,6-Trinitrotoluene
Tetryl	2,4,6-Trinitrophenylmethylnitramine

A. Tested Concentration Range:

HMX	0.376-188 ug/g
RDX	0.253-127 ug/g
NB	0.197-98.4 ug/g
1,3-DNB	0.242-121 ug/g
1,3,5-TNB	0.215-107 ug/g
2,4-DNT	0.240-120 ug/g
2,6-DNT	0.217-109 ug/g
2,4,6-TNT	0.301-151 ug/g
Tetryl	0.265-133 ug/g

B. Sensitivity: Peak height near the detection limit. (1 mm = 28 arbitrary units on the integrator readout.) Representative chromatogram near the detection limit can be found in Appendix I.

	Peak Height in mm at an Attenuation of 2-2
HMX	12 mm for 0.754 ug/g
RDX	18 mm for 0.506 ug/g
NB	11 mm for 0.394 ug/g
1,3-DNB	23 mm for 0.485 ug/g
1,3,5-TNB	20 mm for 0.430 ug/g
2,4-DNT	16 mm for 0.480 ug/g
2,6-DNT	9 mm for 0.434 ug/g
2,4,6-TNT	19 mm for 0.602 ug/g
Tetryl	10 mm for 0.530 ug/g

C. Detection Limits:

HMX	0.376 ug/g
RDX	0.474 ug/g
NB	0.197 ug/g
1,3-DNB	0.242 ug/g
1,3,5-TNB	0.231 ug/g
2,4-DNT	0.240 ug/g
2,6-DNT	0.217 ug/g
2,4,6-TNT	0.301 ug/g
Tetryl	0.265 ug/g

D. Interferences:

1. Any compound that is extracted from soil that gives a retention time similar to the nitro-compounds and absorbs U.V. at 250 nm.

2. Millipore GFWP-01300 filter type GS pore size 0.22 micrometers dissolve in the solvent used.

3. Tetryl and 2-amino-4,6-dinitrotoluene coelute. If a tetryl peak is found in samples, pH adjustment is necessary to separate the peaks to determine which compound is present.

4. 2,4,6-Trinitrobenzaldehyde decomposes rapidly in water solution. Once the acetonitrile standard is made into mobile phase this becomes a problem.

E. Analysis Rate:

After instrument calibration, one analyst can analyze two samples in one hour. One analyst can conduct sample preparation at a rate of three samples per hour. One analyst doing both sample preparation and the HPLC analysis can run 16 samples in an 8-hour day.

II. CHEMISTRY:

A. Chemical Abstracts Service Registry Number:

HMX	2691-41-0
RDX	121-82-4
NB	98-95-3
1,3-DNB	99-65-01
1,3,5-TNB	99-35-4
2,4-DNT	121-14-2
2,6-DNT	606-20-2
2,4,6-TNT	118-96-7
Tetryl	479-45-8

I. Chemical Reactions:

1. RDX and HMX can undergo alkaline hydrolysis.

2. RDX and HMX degrade at temperatures greater than 80°C in an organic solvent.

II. Physical Properties:

	Formula	Mol. Wt.	M.P. (°C)	B.P. (°C)
HMX	$C_4H_8N_8O_8$	296.16	276	-
RDX	$C_3H_6N_6O_6$	222.12	205	-
NB	$C_6H_5NO_2$	123.11	6	211
1,3-DNB	$C_6H_4N_2O_4$	168.11	90	302
1,3,5-TNB	$C_6H_3N_3O_6$	213.11	122	315
2,4-DNT	$C_7H_6N_2O_4$	182.14	71	300 (decomposes)
2,6-DNT	$C_7H_6N_2O_4$	182.14	66	-
2,4,6-TNT	$C_7H_5N_3O_6$	227.13	82	240 (decomposes)
Tetryl	$C_7H_5N_5O_8$	287.15	131	187

III. APPARATUS:

A. Instrumentation: Perkin Elmer series 4 High Performance Liquid Chromatograph (HPLC) equipped with a Perkin Elmer ISS-100 Auto-Injector and Perkin Elmer variable wavelength detector LC-75. Hewlett Packard 3390 recording integrator in peak height mode was used to record the data output.

B. Parameters:

1. Column: Two columns are used in series, in the order listed.

   a. DuPont Permaphase^{R} ODS guard column.

   b. DuPont Zorbax^{R} ODS 4.6 mm i.d. x 25 cm HPLC column with a particle size of 5-6 microns.

2. Mobile Phase: The water/methanol ratio must be adjusted as described in the calibration Section V C to obtain optimum peak separation.

   44-50% water
   28-34% methanol
   22% acenotrile

3. Flow: 1.6 mL/min with a pressure of approximately 2860 psig.

4. Detector: 250 nm

5. Injection Volume: 50 uL.

6. Retention Times:

	Minutes
HMX	3.38
RDX	4.21
NB	7.33
1,3 DNB	6.63
1,3,5-TNB	5.74
2,4-DNT	9.89
2,6-DNT	9.50
2,4,6-TNT	8.93
Tetryl	7.98

C. Hardware/Glassware:

1. Syringes: 25 uL, 50 uL, 100 uL, 250 uL, 5 mL gas tight syringe (Hamilton 1005 TEFLL)

2. Serum vials with crimp caps and Teflon-lined septa Nominal volume of 0.25 mL, 1 mL, 5 mL.

3. Pasteur pipettes and disposable micropipettes.

4. 13 mm stainless steel syringe filter holder (Rainin Instrument Co., Inc. #38-101)

C. Hardware/Glassware: (continued)

5. 13 mm x 0.5 micron fluorocarbon filter (Rainin Instrument Co., Inc. #38-103 Zefluor disc)

6. Whatman 10 mm glass microfiber prefilter

7. U.S. Sieve series 600 (30 mesh)

8. Aluminum foil pans

9. Liquid chromatograph column 1" o.d. x 12"

10. 2 mL, 3 mL, and 5 mL pipettes

D. Chemicals:

1. Acetonitrile, distilled in glass for HPLC use
2. Methanol, distilled in glass for HPLC use
3. Ethyl Ether, distilled in glass for HPLC use
4. Hexane, distilled in glass for HPLC use
5. ASTM Type II Water
6. SARMs for the nitro-compounds

II. STANDARDS: All concentrations are based on a stock solution concentration of 2000 mg/L. Appropriate adjustments should be made if actual concentration varies from this figure.

A. Calibration Standards:

1. Stock Calibration Standards: Stock solutions containing approximately 2000 mg/L of a nitro-compound are prepared by accurately weighing 10 mg of a SARM into a 5 mL serum bottle and dissolving the nitro-compound in 5 mL of acetonitrile pipetted into the bottle. All compounds appear to be stable for 3 months.

2. Intermediate Calibration Standards: All compounds appear to be stable for 3 months.

1. Intermediate Calibration Standard A (high level): Add the following volumes of stock calibration standard and seal with a Teflon-lined septum cap. Store in the dark @ 0°-4°C. The resulting solution (5.8 mL) will have the concentrations indicated in the following table.

A. Calibration Standards: (continued)

Intermediate Calibration Standard A

Nitro-compound	Amt. (uL) of Stock Cal. Std. to add	Resulting conc. (ug/mL)
HMX	1000	345
RDX	600	207
NB	400	138
1,3-DNB	500	172
1,3,5-TNB	500	172
2,4-DNT	500	172
2,6-DNT	500	172
2,4,6-TNT	700	241
Tetryl	600	207
TNBA*	500	172

*2,4,6-Trinitrobenzaldehyde was originally included for certification. However, the compound is too unstable in water solutions to obtain reproducible certification data. It was included in this table as it affects the total volume used to calculate concentration of the other nitro-compounds.

b. Intermediate Calibration Standard B (low level):

Pipette 4.5 mL of acetonitrile into a 5-mL serum vial. Add 500 uL of Intermediate Calibration Standard A. Seal with a Teflon-lined septum cap and store in the dark @ 0-4°C. The resulting solution (5.0 mL) will have the concentrations indicated in the table below:

Intermediate Calibration Standard B

Nitro-Compound	Resulting conc. (ug/mL)
HMX	34.5
RDX	20.7
NB	13.8
1,3-DNB	17.2
1,3,5-TNB	17.2
2,4-DNT	17.2
2,6-DNT	17.2
2,4,6-TNT	24.1
Tetryl	20.7

A. Calibration Standards: (continued)

3. Working Calibration Standards: To a series of ten 5-mL serum vials, approximately one gram of prepared soil (see section V.B.) is accurately weighed into each vial. Using a syringe, the volumes of intermediate standard solutions indicated in the following table are injected onto soil. The serum vial is covered with a septum and shaken until the soil no longer looks wet (approximately 60 seconds). The septum is removed and the indicated amount (see Table below) of acetonitrile is pipetted onto the soil. The septum is replaced and the cap crimped on the vial. The sealed sample is blended on a vortex mixer for approximately 2-3 minutes. The sample is prepared via the procedure given in this method, to give the target concentrations in the following table.

WORKING CALIBRATION STANDARDS

Cal. Conc.	Amt. (uL) Intermed. Cal. Std. to Add		Amt. (mL) Aceto-Nitrile to Add	Resulting Concentration (ug/g)				
	A	B		HMX	2,4,6-TNT	Tetryl	1,3-DNB; 1,3,5-TNB; 2,6-DNT; 2,4-DNT	NB
B	0	0	2.0	0	0	0	0	0
0.1 X	-	12	2.0	0.414	0.289	0.248	0.206	0.166
0.2 X	-	24	2.0	0.828	0.578	0.497	0.413	0.331
0.5 X	6	-	2.0	2.07	0.145	1.42	1.03	0.828
1 X	12	-	2.0	4.14	2.89	2.48	2.06	1.66
2 X	24	-	2.0	8.28	5.78	4.97	4.13	3.31
5 X	60	-	2.0	20.7	14.5	14.2	10.3	8.28
10 X	120	-	1.9	41.4	28.9	24.8	20.6	16.6
20 X	240	-	1.8	82.8	57.8	49.7	41.3	33.1
50 X	600	-	1.4	207	145	142	103	82.8

B. Control Spikes: Control spikes are prepared in the same manner as the calibration standards.

C. PROCEDURE:

NOTE THE FOLLOWING SAFETY PRECAUTIONS:

1. A 5-mL gas tight syringe (Hamilton 1005 TEFLL) is used as the teflon/glass seal is less likely to cause an explosion than glass/glass.

2. The nitro-compounds are less reactive when wet, so every precaution should be taken to ensure that work areas are kept clean and that solutions are not left unattended and allowed to dry.

3. The filtering apparatus is immersed in a water bath and disassembled under water immediately after use. The danger here is solution getting dried on the threads of the filtering apparatus and detonating.

4. When preparing SARM stock standards from pure compounds which are stored in water, small aliquots are scooped onto a nylon or polyvinylidene chloride filter. The water is vacuum filtered off and an appropriate quantity of the "dried" material is weighed out for stock standard preparation. Any extra compound thus dried is disposed of.

5. Prior to working with explosives, it is advisable to discuss safety/handling/storage requirements with an explosives expert.

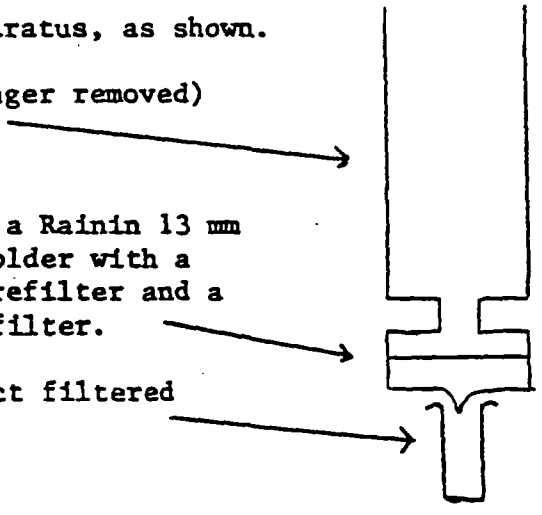
A. Sample Preparation: The soil sample is removed from the sample bottle and spread out in aluminum foil trays. The sample is air dried. The dried soil is screened through a US series 600 sieve (30 mesh). This screened sample is subsampled according to ASTM procedure D346. The moisture content is determined by ASTM Method D2216-71.

B. Extraction:

1. Accurately weigh 1 gram of prepared soil (see section V.A. above) into a 5-mL serum vial, and pipette 2 mL of acetonitrile onto the soil.

   Place a septum and cap on the vial, crimp into place, and shake the vial thoroughly on a vortex mixer for 2-3 minutes.

2. Set up the filtering apparatus, as shown.



f. PROCEDURE: (continued)

3. Prepare the sample for injection as follows:

   a. Pour the sample extract into the syringe.

   b. Place the plunger in the syringe and force at least 500 uL of the filtrate into a 1-mL serum vial.

   c. Using a disposable micropipette, accurately measure 200 uL of filtered extract into a 1-mL serum vial. Accurately measure 600 uL of a 33% methanol/67% water solution onto the filtered sample. This will produce 800 uL of extracted sample in mobile phase.

   d. Place a septum and cap on the vial and crimp into place. Shake the vial well to thoroughly mix. Store in the dark @ 0-4°C until ready to analyze.

4. For samples outside the calibration range, a smaller sample volume is extracted into 5-mL of acetonitrile.

   a. Accurately weigh 0.2 gram of prepared soil into a 5-mL serum vial, and pipette 5 mL of acetonitrile onto the soil. Place a septum and cap on the vial, crimp into place, and shake the vial thoroughly on a vortex mixer for 2-3 minutes.

   b. Prepare the sample for injection as follows:

      1) Pour the sample extract into the syringe.

      2) Place the plunger in the syringe and force at least 3 mL of the filtrate into a 5-mL serum vial.

      3) Using a disposable pipette, accurately measure 1 mL of filtered extract into a 5-mL serum vial. Accurately measure 3 mL of a 33% methanol/67% water solution onto the filtered sample. This will produce 4 mL of extracted sample in mobile phase.

         Alternately, the sample extract and methanol/water solution may be accurately weighed into a 5-mL serum vial. (1 mL $\approx$ 1 g)

4) Place a septum and cap on the vial and crimp into place. Shake the vial well to thoroughly mix. Store in the dark @ 0-4°C until ready to analyze.

c. If the solution prepared from the 0.2 g sample is still above the calibration range, make dilutions of the extract obtained in 4b(1) by taking an appropriate aliquot and adding mobile phase (e.g. 100 mg of acetonitrile sample extract in 20 mL mobile phase) to produce a solution within the calibration range of the instrument.

C. Instrument Calibration/Sample Analysis:

1. Using the auto-injector manufacturer's recommended procedure, introduce 50 uL of the 2X working calibration standard into the chromatographic system. Check the chromatogram to ensure separation of the nitrated toluenes and separation of the nitrobenzene and tetryl. If necessary, adjust the water/methanol ratio of the mobile phase until separate peaks are distinguished. As the column ages, less methanol is required. Generally, the column ages rapidly the first 24 hours, after which it is fairly stable.

2. Once good peak separation is obtained, introduce 50 uL of each working calibration standard and sample into the chromatographic system using the auto-injector manufacturer's recommended procedure.

E. CALCULATIONS:

A. Sample Concentration (ug/g) $= \frac{(\text{peak ht.} - K) \times C \times E}{\text{slope} \times A \times B \times D}$

where:

K = y-intercept of the calibration curve regression line

slope = slope of the calibration curve regression line

$A = \frac{8 \text{ mL mobile phase}}{1 \text{ gram sample}}$ = a constant for this method.

Explanation: the instrument reads the total ug in the 50 uL aliquot of sample injected. This constant enables results to be interpreted as ug/g, as the calibration curve in ug/g is obtained by

$$\frac{2 \text{ mL acetonitrile to extract}}{1 \text{ gram calibration std. sample}} \times \frac{4 \text{ mL mobile phase}}{1 \text{ mL acetonitrile extract}}$$

F. CALCULATIONS: (continued)

B = sample weight

C = mL acetonitrile used to extract sample

D = mL acetonitrile extract diluted into mobile phase

E = final volume in mL of mobile phase prepared for injection

NOTE: When samples are prepared the same as the calibration standards (1 gram extracted into 8 mL of mobile phase), the above calculation becomes:

$$\text{Sample Concentration (ug/g)} = \frac{(\text{Peak height} - K)}{\text{slope}}$$

B. All soils data must be reported on a moisture-free basis. Moisture content is determined by ASTM D2216-71. 100%-% Moisture = % solids.

$$\text{Concentration on a moisture free basis} = \frac{\text{analyte concentration}}{\% \text{ solids}} \times 100$$

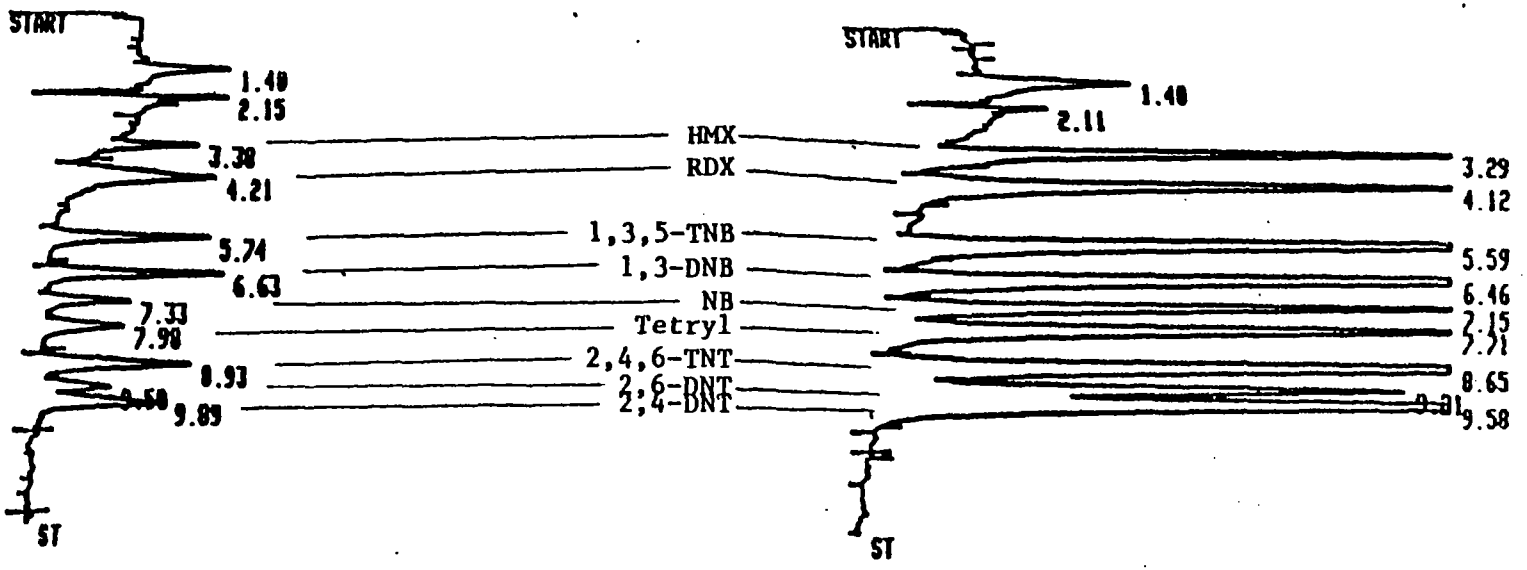
G. REFERENCES:

A. USATHAMA Method 2C Cyclotrimethylenetrinitramine (RDX) in Soil and Sediment Samples, 12-3-80.

B. USATHAMA Method 8H Explosives in Water by HPLC, 12-27-82.

APPENDIX I: CHROMATOGRAMS

EXPLOSIVES IN SOIL - ACETONITRILE EXTRACTION



RUN # 145 APR/22/83 12:58:26
ID 1

HEIGHT%

RT	HEIGHT	TYPE	AR/HT	HEIGHT%
1.40	583	VV	0.435	11.623
2.15	535	PB	0.135	10.666
3.38	332	VB	0.155	6.619
4.21	520	BV	0.365	10.367
5.74	573	PB	0.201	11.423
6.63	632	BP	0.200	12.600
7.33	305	PV	0.208	6.081
7.98	280	VB	0.258	5.582
8.93	539	BV	0.251	10.746
9.58	259	VV	0.243	5.164
9.89	458	VB	0.286	9.131

RUN # 148 APR/22/83 14:04:05
ID 1

HEIGHT%

RT	HEIGHT	TYPE	AR/HT	HEIGHT%
1.40	596	BV	0.344	2.107
2.11	444	PV	0.137	1.570
3.29	2399	PB	0.158	8.403
4.12	2392	BB	0.207	8.458
5.59	4384	PB	0.186	15.502
6.46	4822	PB	0.191	17.050
7.15	2182	BV	0.193	7.715
7.71	2286	VB	0.247	8.083
8.65	3505	BV	0.241	12.393
9.21	1030	VV	0.227	[illegible]
9.58	3441	VB	[illegible]	

ATTACHMENT 2

ANALYTICAL PROTOCOLS FOR DIOXINS AND DIBENZOFURANS

ACKNOWLEDGMENT

[illegible] thanks J. C. T. Hollander for helpful discus-

[illegible] No. H_2SO_4, 7664-93-9.

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[illegible]termination of Part-per-Trillion Levels of Polychlorinated [illegible]benzofurans and Dioxins in Environmental Samples

[illegible] M. Smith,* David L. Stalling, and James L. Johnson

[illegible] *National Fisheries Research Laboratory, U.S. Fish and Wildlife Service, Route 1, Columbia, Missouri 65201*

[illegible]nalytical method permits determinations of parts-per-[illegible] levels and lower of tetrachloro through octachloro [illegible] of dibenzo-*p*-dioxins and dibenzofurans in various [illegible] biological tissues and sediments. Preliminary tests [illegible] indicated the method is applicable to determinations of [illegible] through hexachloro congeners of ortho-unsubstituted [illegible] polychlorinated biphenyls. Interferences both from [illegible] and from xenobiotic substances are reduced to extremely low levels. The procedure has an extremely low [illegible] to false-positive determinations which could result from the presence of a wide variety of cocontaminants. [illegible] approach to contaminant enrichment has permitted [illegible] of seven processes into a two-step procedure, significantly reducing time requirements and the number of [illegible] manipulations, and making the procedure amenable [illegible] automation. The reliability and accuracy of the procedure [illegible] demonstrated by the results of intralaboratory and interlaboratory studies and by successful analyses of over 200 [illegible] of a wide variety of types.

[illegible]chlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and ortho-unsubstituted [illegible] biphenyls (non-ortho PCBs) are three [illegible] and toxicologically related families of anthropogenic chemical compounds that have in recent years been [illegible] have the potential to cause serious environmental [illegible] (*1–6*). These substances are trace-level contaminant byproducts in several large-volume and widely used [illegible] chemicals, principally PCBs and chlorinated phenols [illegible] can also be produced during combustion processes [illegible] and by photolysis (*12*, *13*). In general, PCDDs, [illegible] and non-ortho PCBs are classified as highly toxic [illegible] (*14*), although the toxicities are dramatically dependent on the number and positions of the chlorine substituents (*15*). About 10 individual members of a total of 216 PCDDs, PCDFs, and non-ortho PCBs are among the most toxic man-made or natural substances to a variety of animal species (*1–4*). The toxic hazards posed by these chemicals are exacerbated by their propensity to persist in the environment (*16–18*) and to readily bioaccumulate (*19–21*), and although the rate of metabolism and elimination is strongly species dependent (*20*), certain highly toxic isomers have been observed to persist in the human body for more than 10 years (*22*).

The majority of scientific and governmental concerns for the hazards of these compounds have been directed toward analytical methodologies, toxicology, epidemiology, and determination of the disposition in the environment of the single most toxic isomer, 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) (*1–6*, *8*).

More recently, however, investigations into the formation and occurrence of PCDFs suggest that this family of toxic compounds may commonly occur at comparable or greater levels than and could generally pose a greater hazard than PCDDs. PCDFs are often found as cocontaminants in and are readily produced from pyrolysis of PCBs (*7*, *23–26*). Most important, the PCDFs produced from pyrolysis of PCBs are predominantly the most toxic isomers, those having a 2,3,7,8-chlorine substitution pattern (*5*). A number of recent fires involving electrical transformers and capacitors have demonstrated the potential for formation of hazardous levels of PCDFs from pyrolysis of PCBs (*26–28*, *30*).

In light of these findings and because of the dearth of data pertaining to the occurrence of these compounds in the environment, PCDFs and non-ortho PCBs were included as target compounds in a proposed survey by this laboratory of important U.S. rivers and lakes for PCDDs. The decision to include as many PCDD isomers as possible was based on

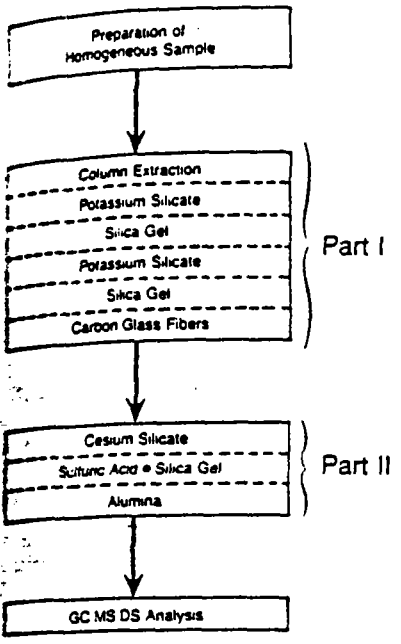


Figure 1. Flow chart of total procedure.

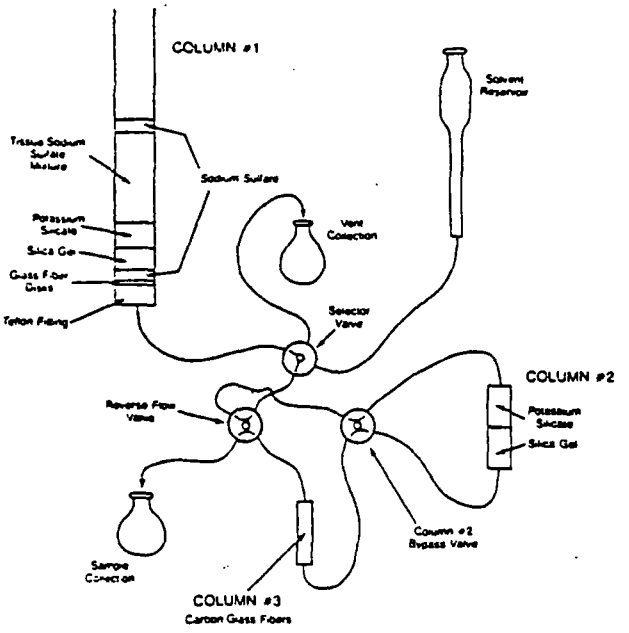


Figure 2. Schematic of part I enrichment apparatus.

toxic (1) several other PCDD isomers are also extoxic (15); (2) pentachlorophenol, a large-volume and wood preservative, contains relatively high levels hepta-, and octachlorodibenzodioxins and essentially TCDDs (7, 8, 29); and (3) incineration of materials conchlorophenols readily produces mixtures of PCDDs, 2,3,7,8-TCDD is a minor component. On the other highly toxic 1,2,3,7,8-pentachloro isomer is a major comof PCDD incineration products of pentachlorophenol Component-specific analyses can be a crucial link to of contamination because different sources of and PCDFs usually produce mixtures of distinctly relative component abundances (7). On the other preferential accumulation of certain isomers in may prevent source identification from analyses of samples.

analytical method developed for this investigation was to meet six criteria: (1) permit determinations of of these compounds, especially those possessing than three chlorine substituents; (2) permit isomerdeterminations of the most toxic or otherwise imcomponents; (3) provide a lower limit of detection for components of between 1 and 5 parts per trillion a variety of environmental samples; (4) generate data acceptable and adequately defined level of accuracy precision; (5) exhibit a very low and well-defined susceptibility to interference and false-positive determinations; minimize analyst's time requirements to permit of large numbers of samples.

determinations of PCDDs and PCDFs demand an unusulevel of analytical assurance, not only because of the hazards of these compounds, the intensity of public concern, and the widespread nature of the but also because of the great difficulties in rigorous of individual isomers. These difficulties are not related to the problems of distinguishing between this problem is essentially solved (31–34)—but are to the possibility of specific and nonspecific interfrom natural and especially xenobiotic substances

Presented herein are the description of an analytical method the results of validation and applications studies which the accuracy and reliability and demonstrate the utility of the method developed for the determination of PCDDs, PCDFs, and non-ortho PCBs in a variety of environmental matrices.

EXPERIMENTAL SECTION

Enrichment Procedure. Tissue and sediment or soil samples (spiked with isotopic marker compounds) are processed in a two-part enrichment procedure (Figure 1). In part I, a mixture of the sample and sodium sulfate is subjected to solvent extraction, and the extract is, in the same process, passed through a series of silica-based adsorbents and then through the carbon/glass fiber adsorbent. The extract passes through the adsorbents in the following order: potassium silicate, silica gel, cesium or potassium silicate, silica gel, and finally an activated-carbon adsorbent. The residues of interest [PCDDs, PCDFs, and non-ortho PCBs, as well as other chemical classes such as polychlorinated naphthalenes (PCNs), polychlorinated biphenylenes, and certain polynuclear aromatic hydrocarbons] are retained on the carbon adsorbent and subsequently recovered by reverse elution with toluene.

In part II of the procedure, following a change of solvent to hexane, the sample is applied to a second series of adsorbents contained in two tandem columns. The first column contains small amounts of cesium or potassium silicate and sulfuric acid impregnated silica gel. The effluent from this column flows directly onto the second column containing activated alumina on which the final fractionation of several classes of residues is accomplished. Following reduction of sample volume, analyses are carried out by high-resolution gas chromatography/low-resolution mass spectrometry/computer data system analysis (HRGC/LRMS/DS) and under some circumstances by gas chromatography/electron capture detector analysis (GC/EC).

Part I. The components of the apparatus used in part I of the enrichment procedure are depicted in Figure 2. Column 1 (about 4.5 cm i.d. and about 1 m long) is connected to column 2 (22 mm i.d. × 100 mm, *Michel-Miller precolumn 5769–34, Ace* Glass, Vineland, NJ) and to column 3 (1.0 cm i.d. × 6 cm thick-walled, precision-bore glass tubing, Kontes, Vineland, NJ) by means of standard $^1/_{16}$ or $^1/_8$ in. o.d. Teflon tubing and tube end fittings. Column 3 is equipped with in-house fabricated Teflon fittings. The solvent flow switching valves are Hamilton minature inert valves (*Hamilton Co., Reno, NV*): selector valve (no. 86781), on–off valve (no. 86775), and bypass and reverse-flow valves (no. 86781). The washing solvent reservoir is constructed of a 20-cm length of 12 mm o.d. glass tubing and a 200-mL reservoir fitted with a 24/40 female ground glass joint. The valving arrangement (Figure 2) is designed to enable the analyst to perform the following operations: venting of the solvent line from column 1, venting of the solvent reservoir, bypass of column 2, delivery of

...from column 1 to columns 2 and 3 sequentially, ...solvent from the reservoir sequentially to columns 2 ...column 3 only, reversal of solvent flow in columns 2 ...stoppage of solvent flow in all lines. The solvent ...routinely pressurized with 1–10 psi nitrogen during ...washings. Column 2 is packed with equal volumes, 15 ...of cesium or potassium silicate and silica gel (EM-60, ...activated) bracketed by plugs of glass wool or preferably ...fiber filters (3-μm retention GF/D, Whatman Inc., ...). Column 3 is packed with a mixture of Amoco PX-21 ...carbon and glass fibers as described previously (*36*). ...packed in the following sequence: two disks of glass ...fibers (GF/D, 4.7-cm diameter, Whatman Inc., Clifton, ...depth of anhydrous sodium sulfate, 30 g of silica gel ...°C activated), 30 g of potassium silicate (130 °C ...50 g of a 1:4 (w/w) mixture of the sample and an... sulfate, and lastly a 2-cm depth of anhydrous ...ce.

...2 (Figure 2) is usually packed with fresh adsorbents ...ple, but this column can be used for more than one ...f the amounts of extracted materials, such as lipids, are ...The carbon adsorbent in column 3 is routinely reused ...washings (under 3–8 psi of nitrogen) between samples ...following solvent sequence: 100 mL each in reverse flow ...methanol, toluene, and cyclohexane/methylene chloride ...A). Column 2 is bypassed during these washings ...the final washing with solvent A, which is directed ...column 2 in the reverse direction to remove residual air ...contaminants. Care must be taken to avoid passing ...through column 2. Another 100 mL of solvent A is ...through columns 2 and 3 in the forward direction ...solvent washing. Complete displacement of toluene ...3 is essential. After columns 2 and 3 are properly ...column 1 is loaded with adsorbents and sample, a ...usually 100 μL) of marker compounds is applied to ...and washed onto the packings with four or five 20-mL ...portions of solvent A using a Teflon wash bottle. ...valve is positioned so that column 1 is connected ...line and air is allowed to escape. The flow of air from ...is monitored as it bubbles through solvent at the vent ...After the sample is spiked with marker compounds, ...of solvent A is carefully applied to column 1, and the ...of the solvent front is observed. As the solvent front ...transfer line (about 1 m in length), air bubbles in the ...removed by stopping the flow and tapping the line. When ...front reaches the selector valve, the valve is reposi... to direct the extract through columns 2 and 3, and the ...procedure is under way. The effluent is collected in ...positioned above columns 2 and 3 to maintain a positive ...on these columns. The height of column 1 above ...flask is adjusted to produce a solvent flow of not ...3 mL/min but sufficient to complete the process ...Occasionally the solvent flow will slow or stop during ...stage and will require the application of 1 or 2 psi of ...to the system at column 1. Rarely, the glass fiber ...inlet end of column 3 become clogged during the ...of decomposed or very oily (especially lake trout) ...To reduce these complications, a removable column (1.0 ...or 3 cm) containing 4 or 5 disks of glass microfibers ...placed in line at the exit end of column 2. If this filter ...becomes clogged, it can be replaced during the process. ...completion of the initial extraction/adsorption op... 3 (bypassing column 2) is washed in the forward ...with 75 mL of solvent A and then 50 mL of methylene ...methanol/benzene (75/20/5) at a flow of approximately ...mL. These washings are collected in the flask with the ...The reservoir is then charged with 40 mL of toluene ...passed through the carbon (column 3) in the reverse-flow ...at approximately 2 mL/min and collected in a 100-mL ...(24/40) flask. At this point, part I of the procedure ...

...in toluene is subjected to rotary evaporation at ...a vacuum of about 0.1–0.2 atm. The rotary evapo... must be maintained in an uncontaminated condition ...washings with organic solvents. No lubricating greases ...The integrity of the sample is protected during rotary evaporation by the use of a vapor trap situated between the sample flask and the evaporation apparatus; the vapor trap is thoroughly washed with toluene between samples. The toluene solution (sample) is carefully reduced to less than 1 mL or just to dryness and removed immediately. The solution or dry sample can be stored in a freezer. At this point, the sample is ready for part II of the procedure (after removal of all toluene).

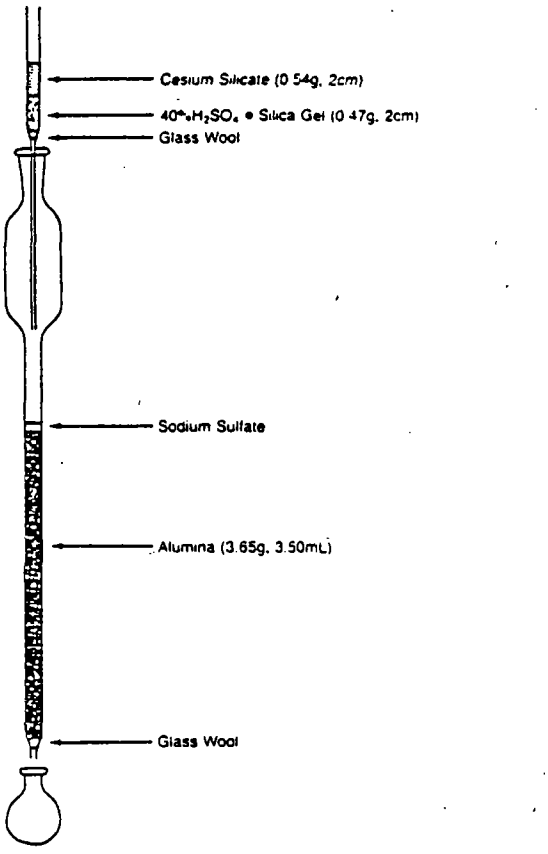


Figure 3. Schematic of part II enrichment procedure.

Part II. The apparatus for part II of the enrichment procedure consists of two columns arranged in tandem (Figure 3). Column 4 is prepared from a disposable Pasteur pipet and is packed first with a plug of glass wool, then with 3 cm (0.50 g) of sulfuric acid impregnated silica gel, then with 3 cm (0.54 g) of cesium potassium silicate (not heat activated), and finally with 0.5 cm of anhydrous sodium sulfate. Column 5 is constructed from a 5-mL graduated pipet fitted with a 20-mL reservoir and a ground-glass joint. Column 5 is packed with a plug of glass wool followed by 3.50 mL (3.65 g) activated (190 °C) alumina and then 0.5 cm of anhydrous sodium sulfate. The alumina is packed firmly by sharply tapping the supporting clamp.

Columns 4 and 5 (Figure 3) are thoroughly washed before use, column 4 with 10 mL of hexane and column 5 under approximately 5 psi of nitrogen pressure, with 30–50 mL of hexane to remove entrapped air. Following the washings, column 4 is partly inserted into column 5 so that the effluent from column 4 flows directly onto the adsorbent bed of column 5. A 50-mL collection vessel is placed at the exit end of column 5. Pasteur pipets previously heated for several hours at 500 °C are used for liquid transfers. The sample is applied to column 4 by using four to six separate 1-mL washings (approximate volumes) of hexane totaling 5.0 mL. Each washing is allowed to pass through column 4 and completely onto the alumina of column 5 before the next washing is applied. After 5.0 mL of hexane has passed through column 4, this column is discarded, and a second 5.0-mL volume of hexane is then applied to column 5. The following sequence of eluting solvents is then applied to column 5: 15 mL of 2%, then 15 mL of 5%, and finally 20 mL of 8% methylene chloride in hexane. A total of 60 mL of effluent is thus collected in two fractions, the first measuring 23 mL and the second 37 mL. Due to variations in the activities of different lots of alumina, the

...tion volumes must be carefully determined for each ...

...second fraction, containing the residues of interest, is ... a volume to about 0.5 mL under a stream of nitrogen ... °C water bath. The sample is transferred to a conical ... with four 0.5-mL washings with methylene chloride, each ... being reduced to a smaller volume under a stream of ... before the next is added. Following the last transfer, ... is completely evaporated and the appropriate volume ... solvent (usually 10 μL of toluene or o-xylene) is added ... to analysis. If the analysis is to be performed later, the ... can be kept in the dry state and stored in a freezer. Before ... is injected, the solution is drawn up into the microliter ... and applied repeatedly to the wall of conical portion of ... to bring the entire sample into solution. Gas chroma- ... spectrometric analyses are carried out by the ... technique (no splitting of the sample) with 2–4-μL ... or by the on-column technique in which 1–2-μL ... are injected.

Sample Preparation. Tissue and sediment samples are ... with at least 4 times their weight of anhydrous sulfate. ... are first cut into small pieces, ground in a meat ... if necessary), and mixed thoroughly with anhydrous ... with a spoon in a glass or polyethylene dish. The ... then spread out to a depth of less than 3 cm so that ... which solidifies after 3–6 h, can be readily broken up ... overnight. The mixture is then dry-blended (any ... blender) in a glass jar to yield a fine powder. ... low water content did not require overnight equili- ... with sodium sulfate before blending. A second blending ... 4–6 h after the first is often required to produce ... homogeneous and finely divided sample.

Analytical Instruments and Conditions. Determinations ... and PCDDs were carried out with a Finnigan 4023 ... system equipped with an INCOS data system and with ... and positive chemical ionization options. Methane was ... the reagent gas for the negative ion chemical ionization ... The gas chromatograph was usually fitted with either ... × 0.25 mm DB-5 fused-silica capillary column (J&W ... Inc., Rancho Cordova, CA) or a 55 m × 0.27 mm Silar ... prepared by H. R. Buser, Swiss Federal Research ..., Wurenswil, Switzerland. The carrier gas was helium and ... following temperature program was routinely used with o- ... 150–255 °C at 3 °C/min and then 12 °C/min to ... and hold for 10 min. The electron impact mode (EI) and ... ion detection (MID) were routinely used for GC/MS ... and quantitation of PCDFs and PCDDs including ... marker compounds ([^{13}C]-TCDD, [^{37}Cl]-TCDF, and ... PCDD). By use of DB-5 column, a series of either 8 or 12 ... charge ratio (m/z) values were monitored within each ... chromatographic windows, each window being defined ... and upper elution limits of a particular group of ... and PCDD congeners. The MID analysis usually involved ... of four or five members of a molecular ion cluster ... of the fragment ion cluster resulting from the ... COCl, M – 63.

... chromatographic analyses employing a packed column [2 ... 3% OV-17 on 100/120 Supelcoport (Supelco, Inc., ..., PA)] were carried out on a Varian 3700 gas chroma- ... equipped with an electron capture detector. Nitrogen ... as the carrier gas with the following temperature pro- ... 270 °C at 8 °C/min and hold for 15 min.

Materials. All solvents were glass distilled grades (MC/B, ..., OH, or Burdick and Jackson, Muskegon, MI). Silica ... 70–230 mesh (EM Reagent, MC/B, Cincinnati, OH) and ... (AG4, Bio Rad Labs, Richmond, CA) were used. ... was washed with methanol and then methylene chloride ... activated at 190 °C for at least 2 days. Silica gel was ... in the same manner and activated at 130 °C. The silica ... stored in the 130 °C oven and removed just prior to use. ... silica (MC/B, no. SX760) is heated at 500 °C overnight ... stored in screw capped bottles.

... PX-21 activated carbon was obtained from the Amoco ... Center, Naperville, IL 60566, and lot numbers 75-8, ..., and 78-10 were successfully used in this laboratory. ... is now commercially available from Anderson Development Co., Adrian, MI 49221, under the name AX-21.

Potassium and cesium silicates were prepared from the reaction of the corresponding alkali metal hydroxides with silica gel in methanol at 55 °C for 90 min. The reaction is carried out in a 1- or 2-L round-bottom flask which is rotated and heated with a rotary evaporation apparatus (no vacuum applied). Sixty grams of CsOH (99+%, Aldrich Chemical Co., Milwaukee, WI) is dissolved in 200 mL of methanol and separated from insoluble material by decantation. An additional 200 mL of methanol is added followed by 100 g of silica gel. For potassium silicate, 168 g of KOH (J. T. Baker Chemical Co., Phillipsburg, NJ), 300 g of silica gel (EM60), and approximately 700 mL of methanol are used; decantation is not necessary for KOH. Following the reaction, the mixture is poured into a large glass column containing a plug of glass wool. Special care must be exercised to avoid contact with the extremely caustic solution, especially eye contact. The adsorbent is washed into the column with methanol, and then 200 mL of methanol for every 100 g of silica gel is added to the column. The methanol can be pushed through the column under slight gas pressure, and as the level of the liquid reaches the bed of adsorbent, 200 mL of methylene chloride for every 100 g of silica gel is applied. The liquid is pushed through the column and the silicate partly or completely dried under a slow flow of nitrogen. Cesium silicate is dried completely under a stream of nitrogen and is not heat activated; potassium silicate is activated at 130 °C.

Sulfuric acid impregnated silica gel (40% w/w), abbreviated as SA-SG, is prepared by adding 2 parts of concentrated sulfuric acid to 3 parts by weight of 130 °C activated silica gel in a screw capped bottle and shaking until the mixture is completely free of lumps, about 15 min. The silica gel is activated at 130 °C; unactivated silica gel is unsatisfactory for the preparation of SA-SG. The adsorbent is stored in a screw capped bottle.

Nitrogen gas used for evaporations of solvents is passed through a copper tube (40 mm o.d. × 60 cm) containing activated carbon (coconut charcoal, Fisher Scientific Co., Pittsburgh, PA) bracketed by glass wool and glass microfiber filters. Following the carbon trap, a microfiber filter (Microfibre filter 9802-AAQ, 505-AAQ, 0.3-μm retention, Balston Filter Products, Lexington, MA) is inserted in the line in an attempt to prevent movement of carbon particles through the nitrogen line.

RESULTS AND DISCUSSION

Development and Functions of the Components of the Enrichment Procedure. *Part I.* A primary objective in the initial approach to the development of this method was to make optimum use of the highly selective absorbtivity of activated carbons for polychlorinated polycyclic aromatic compounds (*37*). The carbon adsorbent selected for this procedure was Amoco PX-21 dispersed in glass fibers (CGF) which has been thoroughly evaluated in this laboratory with regard to its selectivity for a wide variety of chemical classes (*36*, *37*). At least four lots of PX-21 carbon have been successfully employed by this and other laboratories (*26*, *38–46*) in analyses of PCDDS and PCDFs.

Application of extracts of whole fish directly to the carbon absorbent dispersed in glass fibers was found to be generally unacceptable due to the adsorption of biogenic substances causing high back pressures. Pretreatment of the tissue extract with the strongly basic absorbent potassium silicate (KS) (*47*, *48*) followed by activated silica gel (SG) greatly facilitated the flow of the tissue extract through the carbon adsorbent. Other combinations with alumina and with Florisil or with potassium silicate alone were less effective. The combination of KS, SG, and PX-21 carbon adsorbents achieved a very high degree of enrichment of PCDDs, PCDFs, and non-ortho PCBs. Tissue samples up to 50 g and containing 10–20 g of fat routinely give only submilligram residues in the sample recovered by reverse elution of the carbon with toluene. Integration of these three steps yielded a procedure that permitted *simultaneous sample extraction, removal of acidic and highly* polar coextractables, and selective adsorption of the compounds of interest onto carbon (part I) and was readily

...ular arrangement which simplified sample, ...sorbent manipulations (Figure 2). Several sets ...uld each be loaded with a sample, the three ...and solvent, and the enrichment processes allowed ...extended, by gravity solvent flow. The use of ...ation of potassium silicate and silica gel ...ure 2) ensures that the interfering lipid materials ...from reaching the carbon and permits the an... estimate the amount of colored lipid material ...sorbed by the potassium silicate/silica gel ...In those cases in which little or no accumulation ...terial is observed on column 2, consideration can ...using column 2 for another sample. Cesium ...acidic compounds more effectively than KS ...ally used in column 2 but is 50 times more costly. ...operations of part I eliminate the need for ...require extensive sample manipulations and ...labor intensive. Such procedures which are ...employed in other methods include one or more ...(1) acidic or basic digestion of the sample, ...liquid–liquid partitioning steps, (3) Soxhlet ex... gel permeation chromatography. The ability ...enrichment procedures in a one-step, con... can result in enhanced recovery and pre... to reduced analysis time. Furthermore, this ...itself to the possibility of development into ...multisample procedure (*49*).

...chromatography (GPC) was initially em... procedure as an enrichment step preceding the ...but often did not have the capacity for the ...required in these analyses. Furthermore, the ...of GPC into the initial enrichment procedure ...additional sample extraction and solvent volume ...steps precede the GPC procedure.

...to protecting the adsorptive capacity of the ..., the silicate adsorbent has been demonstrated ...to remove acidic compounds which represent ...interferences to determinations of PCDDs ... The silicate adsorbents retain substances which ...acidity constants of 10 and lower, including ...carboxylic acid compounds and sulfonamides ...particular, hydroxy PCBs and hydroxydiphenyl ...ethers which can produce false-positive GC/MS ...effectively removed by the silicates (*35*).

...conditions of this enrichment procedure, the ...will retain only a limited number of classes ...compounds (*50*), including polyhalogenated planar ...matic compounds, to some extent PAHs with ...three rings, and strongly acidic compounds that ...sequestered by the silicate adsorbent before ...carbon. The large majority of synthetic organic ...which are commonly encountered as persistent ...contaminants are weakly adsorbed and readily ...the carbon by the extraction solvent. Included ...of chemicals are compounds which interfere in ...minations of PCDDs, PCDFs, and non-ortho ...DDE, PCBs, methoxy PCBs, polychlorinated ...(PCDPEs), and methoxy PCDPEs (*35*). The ...also exhibits a very low affinity for the ...which are not retained by the potassium ...gel combination.

...part II of the enrichment procedure (Figure 3) ...first passed through a strongly basic adsorbent, ..., and a strongly acidic adsorbent, 40% sulfuric ...silica gel (SA-SG), in the nonpolar solvent, ...subjected to chromatography on acid alu... ...of the sample to cesium silicate in the ...hexane virtually assures the removal of trace residues of acidic compounds. Use of cesium silicate which has been activated at 130 °C resulted in poor recoveries of hepta- and octachloro isomers. The adsorbent should simply be purged of solvent under a stream of nitrogen after preparation and not oven activated.

The sulfuric acid impregnated silica gel (40% w/w) has been demonstrated in this laboratory and elsewhere (*51*) to strongly retain or undergo chemical reactions with a number of classes of compounds. A series of polynuclear aromatic hydrocarbons (PAHs) possessing two to four condensed rings was found in this laboratory to be effectively retained by this adsorbent. The adsorbent is also undoubtedly very effective in removing numerous types of compounds by reactions of dehydration, acid-catalyzed condensations, and oxidation as demonstrated by the complete charring and polymerization of tissue extracts applied to this material. Colored bands of adsorbed materials are normally observed on the SA-SG adsorbent following sample application in part II of this procedure. The reactivity of this adsorbent toward PAHs is complementary to the activated-carbon adsorbent which strongly adsorbs certain PAHs which are subsequently recovered with the PCDDs, PCDFs, and non-ortho PCBs. Because polynuclear aromatic hydrocarbons will elute from alumina under the solvent conditions employed in the subsequent step involving alumina chromatography, it is important that PAHs be removed prior to this step. In some environmental samples, especially sediments, high concentrations of PAHs were frequently encountered.

The final step of the enrichment procedure, alumina chromatography, is designed primarily to separate PCDDs, PCDFs, and non-ortho PCBs from polychlorinated naphthalenes (PCNs), trace residuals of PCB isomers, and other polychlorinated aromatic compounds. In addition to PCDDs, PCDFs, and non-ortho PCBs the only classes of compounds which have been shown in this laboratory and elsewhere (*46*) to be recovered from the carbon are PCNs, polychlorinated biphenylenes, and certain polychlorinated PAHs. The alumina chromatography removes the large majority of the 75 possible PCN isomers, but four to six penta- and hexachloronaphthalenes are partially recovered with the PCDDs, PCDFs, and non-ortho PCBs. Use of basic alumina (190 °C activated) requires higher concentrations of methylene chloride to recover PCDDs and PCDFs.

In-House and Extralaboratory Evaluations and Validation Studies. The following studies and evaluations were made: (a) determinations of the mean recoveries of a series of representative compounds of the three chemical groups at selected concentrations, (b) determinations of the coefficient of variation associated with each set of recovery data, (c) estimation of the lower limit of detection and determination of the various congener groups or individual components in a variety of sample types, (d) evaluation of the degrees of interference posed by seven series of polychlorinated aromatic compounds which represent the greatest threat of producing false-positive data, and (e) determination of the success rate for completed analyses of approximately 200 environmental samples.

Recovery Studies. Initial recovery studies were performed by using an abbreviated procedure which did not incorporate either the silica gel in part I or the alumina chromatography in part II. This procedure was highly effective for the determination of PCDDs, PCDFs, and non-ortho PCBs in biological materials. The major disadvantage of this abbreviated procedure appeared to be the inclusion of a large number of polychlorinated PAHs such as PCNs in the analyte. Nevertheless, an abbreviated procedure excluding alumina chromatography has been successfully used in the analyses of over 30 environmental samples. PCNs were the most significant cocontaminant observed but did not interfere in the deter-

...coveries of Selected PCDDs and PCDFs in Salmon Oil from Abbreviated Procedure: Potassium Silicate, ... Fibers, Cesium Silicate, and Sulfuric Acid-Silica Gel[a]

	recoveries of selected compounds						
	2,3,6,8-Cl_4-furan	2,3,7,8-Cl_4-dioxin	1,2,4,7,8-Cl_5-furan	1,2,3,4,7,8-Cl_6-furan	1,2,3,4,6,8,9-Cl_7-furan	OCDD	OCDF
...ount of ...mple	109 [1]	115 [1]	115 [1]	113 [1]	117 [1]	86 [1]	79 [1]

	recoveries of selected compounds						
	2,3,7,8-Cl_4-furan 2,3,7,8-Cl_4-dioxin[b]	1,2,4,7,8-Cl_5-furan	1,2,4,6,7,9-Cl_6-furan	1,2,3,4,7,8-Cl_6-dioxin	1,2,3,4,6,8,9-Cl_7-furan	OCDD	OCDF
...ount of ...mple	81 (9) [4]	70 (5) [4]	75 (5) [4]	82 (3) [4]	77 (5) [4]	87 (7) [4]	75 (5) [4]
...ch in ...d oil	102 (2) [4]	97 (3) [4]	84 (4) [4]	98 (2) [4]	87 (6) [4]	76 (3) [4]	74 (5) [4]
...ch in ...d oil	66 (2) [3]	80 (–) [2]	68 (3) [3]	76 (–) [2]	72 (8) [3]	66 (3) [3]	62 (14) [3]

...ons were determined on a 12-m OV-17 WCOT glass column and electron capture detection (^{63}Ni) using helium at 50 cm/s and the ...perature program: 190 °C for 2 min, then 4 °C/min to 240 °C and hold 15 min. Numbers in parentheses are coefficients of ... Numbers in brackets are the number of replicate samples analyzed. [b] 2,3,7,8-TCDD and 2,3,7,8-TCDF coeluted on the OV-17

...coveries of Selected PCDDs and PCDFs from Spiked Samples of Homogenized Whole Fish Using the ...ed Enrichment Procedure

	recoveries of selected compounds							
...mple	2,3,6,8-Cl_4-PCDF	2,3,7,8-Cl_4-PCDF and PCDD	1,2,4,7,8-Cl_5-PCDF	1,2,4,6,7,9-Cl_6-PCDF	1,2,3,4,7,8-Cl_6-PCDD	1,2,3,4,6,7,9-Cl_7-PCDF	OCDD	OCDF
...of ...carp and ...ch of PCDD ...CDF (100 pptr)	81 (1) [4]	92 (3) [4]	94 (3) [4]	98 (6) [4]	104 (4) [4]	95 (8) [4]	99 (22) [4]	91 (16) [4]

	recoveries of selected compounds			
sample	[^{13}C]-2,3,7,8-TCDD	[^{37}Cl]-2,3,7,8-TCDF	[^{37}Cl]-1,2,7,8-TCDF	[^{37}Cl]-OCDD
...mples spiked at 25–50 pptr	82 ± 27 [49]	58 ± 16 [11]	75 ± 18 [10]	83 ± 30 [18]

	recoveries of selected compounds[a]								
	Cl_4 PCDFs	Cl_5 PCDFs	Cl_6 PCDFs	Cl_7 PCDFs	OCDF	Cl_5 PCDD	Cl_6 PCDD	Cl_7 PCDD	Cl_4 biphenylene
...ked at 20 pptr	58 ± 10	64 ± 6	64 ± 7	63 ± 10	59	41	49	58	52
...ked at 100 pptr	52 ± 7	55 ± 4	53 ± 6	56 ± 4	52	84	60	51	59

...of PCDDs and PCDFs. The recoveries of a series ... and PCDFs from spiked samples of salmon oil by ... abbreviated procedures are given in Table I. ... of spiked fish samples containing up to 20 g of oil ... carried out by GC/EC and showed very low levels ... to less than 50 pptr for the most prominent ... of matrix components in the analytes (*49*). ... incorporation of silica gel in part I and alumina ... I of the procedure, recoveries of a series of PCDDs ... from spiked whole fish samples were again de- ... (Table II). Recently, an independent evaluation ... procedure was carried out at the University ... laboratory and included the determinations of re- ... spiked fish of a mixture of fourteen tetra-, five ... hexa-, three hepta-, and one octachlorodibenzo- ... penta-, one hexa-, and one heptachlorodibenzo- ... and one tetrachlorobiphhenylene (*45*). Mean and ... deviations of the recoveries are presented herein to ... support the effectiveness of the method for the con- ... groups.

...substituted PCBs have been detected in about ... of the environmental samples analyzed by this method. Only two sets of recovery determinations have been made for three representative non-ortho PCBs spiked at 100 ppb: 3,4,3′,4′-tetrachloro (38 and 57%), 3,4,5,3′,4′-pentachloro (43 and 47%), and 3,4,5,3′,4′,5′-hexachloro (54 and 59%).

The demonstration of the effectiveness of recovery of a large selection of PCDD and PCDF isomers, in particular those tetra-, penta-, and hexachloro isomers possessing the critical 2,3,7,8-chlorine substitution pattern, is especially important to defining the comprehensiveness and applicability of the method. The recoveries of all the isomers studied are generally comparable and no particular isomer or group of isomers appear to be selectively excluded by the enrichment procedure.

In addition to the recovery data derived from spiked samples as part of the validation studies, a substantial collection of recovery data was also generated for the four major components of the marker compounds which were added to each sample prior to the enrichment process. The marker compounds, [UL-^{13}C]-2,3,7,8-TCDD, [UL-^{37}Cl]-OCDD, and a mixture of six [UL-^{37}Cl]-TCDFs including [^{37}Cl]-1,2,7,8- and [^{37}Cl]-2,3,7,8-TCDFs as the major components, were routinely incorporated into each sample at levels of 50, 50, 25, and 25 pptr, respectively. Although the range of recovery data values

...lative Recoveries of Tetrachlorodibenzo-*p*-dioxins from the Unabbreviated Enrichment Procedure[a]

...MS ... no.	TCDD isomer	rel recovery	GC/MS peak no.	TCDD isomer	rel recovery
1	1368	1.20	8	1234, 1237, 1238, 1246, 1249	1.45
2	1379	1.27	9	1236, 1279	1.47
3	1378	1.57	10	1278, 1469	1.35
4	1369, 1247, 1248	1.47	11	1239	1.39
5	1268	2.13	12	1269	1.39
6	1478	1.30	13	1267	2.85
7	2378	1.00	14	1289	3.62

[a] ...approximately 2 ng of TCDDs was applied i.d. the enrichment procedure. Determination was made on a 60 m × 0.25 mm i.d. ..., Inc.) capillary column under MID-EI mass spectrometric conditions: temperature, 200 °C for 1 min, then to 250 °C at 5 ... and held; He carrier gas.

...marker compounds generally reflects the reduced ... GC/MS/DS quantitation of trace analytes using ... standard technique, the determinations of the ... of the marker compounds in these samples per... nearly a 3-year period provide a practical measure ... performance of the enrichment procedure and the ... analytical method (Table II). The average recoveries ... for the marker compounds over this extended period ... to be consistently satisfactory with the exception ... [^{37}Cl]-2,3,7,8-TCDF which in early studies was ob... to be uniformly low in comparison with those of the ... marker compounds, most conspicuously with those of ... other [^{37}Cl]-TCDFs. A reexamination of the elution ... 2,3,7,8-TCDF from alumina suggested that this step ... was sources of the problem; 2,3,7,8-TCDF eluted very ... the collection cutoff point. The addition of 5 mL to ... elution volume increased the recovery of [^{37}Cl]-... TCDF to levels comparable with those of the other ... compounds.

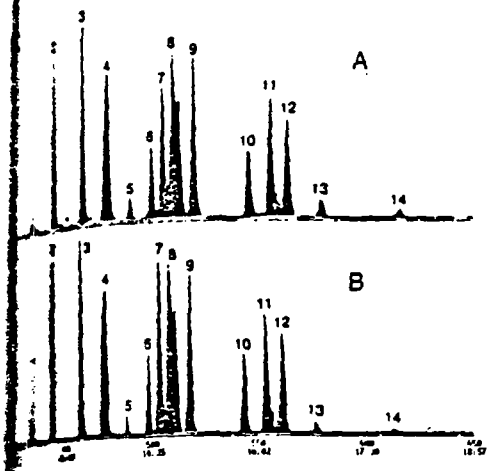
...ations of background levels of PCDDs, PCDFs, ... non-ortho PCBs were routinely made as part of the ... control protocol. Procedureal blanks and samples of ... laboratory-reared fish, each spiked with the ... compounds, were incorporated at a frequency of about ... in all sample sets. Analyses of these control samples ... define the background level for sample sets and to ... possible residue carry-over among samples. Of 14 ... blanks, 1 produced a positive determination for ... CDD at 1.6 pptr, 7 were positive for OCDD (1, 5, 7, ... and 11 pptr), 1 was positive for a 2,3,7,8-TCDF at 2 ... and 2 were positive for OCDF at 0.5 and 1.4 pptr. All ... results for the 10 congener groups (total of 140 deter... in these procedural blanks were negative and were ... by an average lower limit of detection of ap... 2 pptr. Of 11 analyses of samples of laborato... carp, 7 produced positive determinations for ... 5, 7, 18, 24, and 39 pptr), 7 were positive for ... CDF (1, 1.5, 2, 3, 3, 3, and 6 pptr), 1 was positive for ... 4 pptr, 1 for a HCDF at 2 pptr, 3 for a HpCDF ... pptr), and 5 were positive for OCDF (1, 1, 2, 3, ... The remainder of the 110 determinations of ... PCDFs in these control fish were negative. The ... of detection was approximately 2 pptr. Non... were not observed in these control samples, and ... limit of detection for these compounds was ap... 5 pptr. In one series of control samples of lab... trout, a number of PCDF isomers were re... at 10–20 pptr levels. These compounds were ... as trace contaminants in the commercial fish ... in the rearing.

...background levels of PCDDs, PCDFs, and non... were negligible, especially for those isomers ... the 2,3,7,8-substitution pattern. Octachlorodi... appears to be a common trace environmental contaminant, being detected in more than 50% of the fish samples at levels significantly above those observed in the procedural blanks.

Although repeated analyses of procedural blanks between sample sets established a nondetectable level of carry-over between biological samples containing widely varying concentrations of PCDDs, PCDFs, and non-ortho PCBs, sample cross-contamination (from a carbon column) was observed to result from certain types of samples containing abnormally high levels of these contaminants. The samples causing cross-contamination were pond and river sediments and a sample of Aroclor 1260, all containing relatively high concentrations of PCDFs. Carry-over of PCDFs was readily demonstrated to result from reuse of the carbon columns and was observed in samples of fish which were processed on the same carbon column used for the highly contaminated samples. The degree of carry-over appeared to be on the order of 0.1%. In general, procedural blanks should be incorporated in sample sets at a frequency which will permit early detection of carry-over problems and should be included immediately following samples suspected of containing abnormally high concentrations of PCDDs, PCDFs, and non-ortho PCBs. Particularly in the case of sediment samples, high levels of other types of contaminants are routinely encountered, especially polynuclear aromatic hydrocarbons, and saturation of the carbon adsorbent with these substances may contribute to the problem of carry-over of PCDDs and PCDFs. In two cases of gross contamination of the carbon adsorbent, repeated washings of the column did not completely eliminate the problem, and the columns were replaced.

A satisfactory and reproducible level of recovery for 2,3,7,8-TCDD having been established, the recoveries of the other 21 TCDD isomers were examined. The mass chromatograms of a mixture of the 22 TCDD isomers (mixture provided by Dr. H. R. Buser, Swiss Federal Research Station, Wadenswil, Switzerland) before and after having been subjected to the enrichment procedure are presented in Figure 4. The relative recovery data, normalized to the recovery of 2,3,7,8-TCDD, are given in Table III. These data, although not rigorously demonstrative of satisfactory recoveries for each of the other 21 isomers, do establish that most of these isomers were effectively recovered by the procedure. In fact, in this experiment all other isomers or groups of isomers were apparently recovered more efficiently than was 2,3,7,8-TCDD. The abnormally high calculated recoveries of the 1,2,6,8-, 1,2,6,7-, and 1,2,8,9-TCDDs, each a minor component of the mixture, are attributed to the disproportionate influence of variations in instrumental sensitivity on analyte response near the limit of quantitation.

Probably the most useful piece of information derived from an examination of the determinations of the marker compounds in the hundreds of samples was the fact that the success rate for analyzability of the samples was better than 99% and that the minimum level of detection consistently



...MS-MID electron impact ion chromatograms of 22 ... (A) following application of enrichment procedure; (B) ...

... of 1–10 pptr with an average value of less than ... samples and controls were routinely spiked at the ... level with each of the marker compounds. In all ... indicated by the positive and uniform responses of ... compounds in each of the analytes, GC/MS ... CDDs and PCDFs at low parts-per-trillion levels ... attainable. Estimates of the lower limit of ... (LOD) for TCDDs, TCDFs, and OCDD were made ... from the observed signal-to-noise value for ... marker compounds (internal standards) to the ... corresponding to a signal-to-noise value of 3. ... LOD require comparisons of the noise levels in ... of each group of compounds and appropriate ... of the internal standards.

... of PCDDs, PCDFs, and non-ortho PCBs ... increasingly difficult at levels approaching the limit ... due particularly to increased variations in the ... of the isotopic components of the molecular ... requirement of the correct isotopic abundance ratios ... ions in determinations of PCDDs and ... at low parts-per-trillion levels was usually the most ... to meet once sufficient instrumental sen... attained. Nevertheless, over 50 separate confir... made of PCDD and PCDF residues present at ... pptr. The criteria for the confirmation of any ... or non-ortho PCB of unspecified substitution ... (1) signal-to-noise ratio of ≥3; (2) correct ... mass; (3) coincidental maxima of three or ... scans of individual members of the molecular ... and (4) chlorine isotope ratios within 10% ... values for three to six members of the molecular ...

... of routine monitoring of the fragment ions ... characteristic loss of COCl from PCDDs and ... investigated and determined to be marginal for ... at part-per-trillion levels due to the relatively ... for these ions. The criteria for confirmation of ... also include a requirement of demonstrating ... unique relative retention time within 2–4 parts ... example, 2,3,7,8-TCDD is sufficiently resolved ... TCDD isomers on both a Silar 10C (31) and ... Inc.) (52) capillary column to enable easy ... of acceptable limits for the variation in re... this isomer relative to that of the isotopic ...-2,3,7,8-TCDD. The retention time of ... on the DB-5 column was also found to be ... partial overlap with the 1,2,3,7- and ... indicated that their presence could be obscuring

but would not produce a false-positive determination. The variation in the retention time of 2,3,7,8-TCDD relative to that of [^{13}C]-2,3,7,8-TCDD on the DB-5 column was observed in numerous analyses of standard mixtures of the two compounds and found to be within 2 parts in 1000. All confirmations of 2,3,7,8-TCDD in samples analyzed by this procedure met this requirement and were often repeated on a Silar 10C column. Samples of particular importance were independently analyzed by other laboratories using complementary techniques such as high-resolution mass spectrometry or atmospheric-pressure chemical ionization mass spectrometry (53). Over 20 samples analyzed in this laboratory for PCDDs and PCDFs were subjected to independent analyses in other laboratories, including those of H. R. Buser (Switzerland Federal Research Station, Wadenswil, Switzerland) (54), Ronald Mitchum (National Center for Toxicological Research, Jefferson, AR) (55), Michael Gross (University of Nebraska, Lincoln, NB) (55), Robert Harless (USEPA, Research Triangle Park, NC), David Firestone (U.S. Food and Drug Administration, Division of Chemistry and Physics, Washington, DC) (56), John Ryan (Health and Welfare Canada, Food Division, Ottawa, Canada) (57), Patrick O'Keefe (New York State Department of Health, Albany, NY) (26), and Christopher Rappe (University of Umea, Umea, Sweden) (Table IV). The Columbia laboratory also participated in three interlaboratory studies of the effectiveness of different methods for the determination of 2,3,7,8-TCDD in fish. The agreement in both identification and quantitation between the results from this laboratory and those of the other laboratories was consistently good, and no false-positive results were indicated in any of the determinations made with this procedure (Table IV). In the majority of interlaboratory studies, the comparisons involved only determinations of 2,3,7,8-TCDD.

Evaluation of Potential for Interference from Cocontaminants. Determinations of PCDDs, PCDFs, and non-ortho PCBs in environmental samples at levels below 1 pptr are particularly susceptible to interferences and possible false-positive results as a consequence of the likely occurrence of a large variety of polychlorinated aromatic cocontaminants and because full-scan mass spectrometric analyses are usually unattainable. More than a dozen families of such compounds are recognized as potential interferences in these types of analyses (35, 58), including DDE and DDT and polychlorinated members of the following compounds: biphenyl (59), methoxybiphenyls (60), hydroxybiphenyls, diphenyl ether (61), methoxydiphenyl ethers, hydroxydiphenyl ethers (62), benzyl phenyl ether (63), naphthalene, biphenylene, phenylbenzoquinone (64), xanthene, and bis(phenoxy)methane (65). Most of these families of compounds have the potential to interfere with and produce false-positive results in determinations of PCDDs and PCDFs even in HRMS (35). The problem of interferences in determinations of PCDDs and PCDFs has been rigorously addressed experimentally in only a few publications (66), and in these was limited to a small proportion of the numerous families of potential interferences. Routinely, conclusions in regard to the potential for interferences in analytical procedures for PCDDs and PCDFs are made by inference from observations of the effectiveness of separation of comparable amounts of these interfering compounds from PCDDs and PCDFs, often with a relatively small number of isomers of these two families. For example, alumina has been shown to effectively separate PCBs from certain PCDD isomers (67). A more appropriate evaluation should include a large number of isomers of and a large excess concentration (10^4–10^6) of the potential interference relative to that of PCDDs or PCDFs.

As part of the validation of this procedure an evaluation was made of the degrees of interferences produced by seven

...sults of Interlaboratory Studies and Comparisons of the Determination of 2,3,7,8-TCDD in Fish and Birds

		levels of 2,3,7,8-TCDD reported (pg/g) at different laboratories							
...udy	CNFRL	no. 1	no. 2	no. 3	no. 4	no. 5	no. 6	no. 7	reported av
...[a]	9					6	5		
...ample 1	47	67			77	89	67		
...ample 2	22	25			57	42	34		
...ample 3	117	113		b	128	99	188		
...ample 4	56	45	b	b	38	53	c		
...ample 5	96	100	b	b	107	199	178		b
...ample 6 (USFDA[d])									
...ample 7	58	104	58	49, 58	<5	72	70	60	61
...ample 8	<1	<10	<1	<2, <2	<5	<2	<5	37	3.6
...ample 9	34	35	37	23, 32	51	25	33	26	30
...ample 10	38	45	33	19, 31	55	32	27	32	32
...[e]									
...ample 11	37	52	45	55					
...ample 12	36	39							
...ample 13	19	15	25						
...ample 14	<1	<9	<5	<25					

Independent Laboratories

	CNFRL	Swiss Fed Res[f]	Nat Center Tox Res[g]	Health & Wel Can.[h]
...ring gull, Lake Huron	160	165		132
...gull egg, Detroit River	70	75		80
...rp, Lake Huron	22, 27	29	10	
...rp, Lake Erie	<1	5	<10	
...ake trout, Lake Ontario	56, 58		54	
...herring, control	<1		<10	
...ake trout, Lake Huron	39		32	
...ch trout, Lake Ontario	38		31	
...rp, Saginaw Bay	94		75	
...rp, Tittabawassee R., MI	81		65	

...ed. [b]Samples were not analyzed due to large amounts of materials in analyte. [c]Sample was lost. [d]Reference 50. [e]Reference ...MS EI. [f]HRGC/MS API. [g]HRGC/HRMS EI.

...polyhalogenated aromatic compounds (*35*). In... the study were selected isomers of polychlorinated ...PCBs), naphthalenes (PCNs), diphenyl ethers ...methoxybiphenyls (MEO-PCBs), methoxydi... ethers (MEO-PCDPEs), hydroxybiphenyls (HO... and hydroxydiphenyl ethers (HO-PCDPEs). The ...ses an upper limit to the level of interference ...these individual compounds. The results dem... ability of the procedure to effectively eliminate ...from all but a small number of PCN isomers and ...except non-ortho) present at concentrations of ...of those of the PCDDs and PCDFs. Levels ...200–500 000 times those of PCDDs and PCDFs ...ly observed in environmental samples analyzed ...tory (*68*), but PCB isomers other than the ...PCBs have not been observed in the analyses for ...PCDFs. Furthermore, the results suggest that ...is not susceptible to interference from 10 000- ...of the other five families of compounds. About ...mers are recovered by the procedure and are ...served in environmental samples but do not ...positive determinations. Rarely, interference ...ation procedure due to partial overlap of a Cl_6 ...with the marker compound, [UL-^{13}C]-2,3,7,8-T...ered. The effective elimination of numerous ...chlorine compounds, such as DDE, known to be ...of the fish samples which were analyzed by ...has been demonstrated by full-scan MS ...

...also recovers isomers of polychlorinated ... A large number of isomers of polychlorinated ...were identified in this laboratory in a sample of soot produced during an electrical accident involving the pyrolysis of PCBs in a state office building in Binghamton, NY, in 1982 (*26*, *69*).

The only other group of polychlorinated aromatic compounds apparently observed in a small percentage of samples were the nonachloromethoxydiphenyl ethers. These compounds, of which there are three possible isomers, were tentatively identified in three fish samples, from Saginaw Bay (*35*, *68*), the Housatonic River, and Chesapeake Bay. The presence of these cocontaminants in the analyte contrasts with studies of interferences which indicate that chlorinated methoxydiphenyl ethers would readily be separated from PCDDs, PCDFs, and non-ortho PCBs.

The presence of polychlorinated diphenyl ethers (PCDPEs) in the analyte can be especially problematic because these compounds often undergo fragmentation during electron impact MS by loss of two chlorines to produce mass spectra which are identical with those of PCDFs below the molecular ion of the diphenyl ether. Furthermore, the elution window of PCDPE congeners have been observed in this laboratory to overlap that of PCDF congeners possessing two less chlorine substituents, greatly increasing the possibility for false-positive determinations from GC/MS-MIM analyses. Monitoring of masses of the molecular ions of the PCDPEs, if practical, can essentially eliminate this possibility.

The susceptibility to interferences of these types of analyses is demonstrated by the results of an interlaboratory study conducted by the USFDA (*56*) of the effectiveness of six different enrichment procedures (for 2,3,7,8-TCDD) performed by six independent laboratories (see Table IV). The enriched samples were all returned to the USFDA laboratory for rigorous analysis. Of the seven sets of analytical results only two,

Precision of Quantitation Using Internal Standards in GC/MS and GC/EC Analyses Before and After the Procedure

	before enrichment procedure			after enrichment procedure		
	mean response by GC/MS[b]	% std dev by GC/MS[b]	% std dev by GC/EC[c]	% std dev by GC/MS[b]	% rel recovery by GC/MS[b]	% rel recovery by GC/EC[c]
	1.39	6	8	12	97	109
	0.54	17	5	8	96	113
	1.40	7	2	14	97	128
	0.05		2		160	129
	1.05	7		5	85	
	0.92	15	4	19	140	127
	1.36	19	10	9	80	107
	5.63	10	2	15	109	126
	1.60	9	4	16	113	150
	1.29	9	5	17	133	137
	1.18	5	5	17	143	150
	0.80	8	7	15	153	141
	0.97	11	4	20	135	157
	0.42	12	8	26	195	159
	0.31	26	7	36	177	114
	0.44	27	7	30	164	114
	0.18	5		5	117	
	0.88	10		13	103	
	0.97	6		14	78	
	1.00				100	
	0.66	18		18	115	
		11.9	5.1	16.3		
		9.8	4.9	14.1		

including Cl_8

D = PCDD. [b] [13C]-2,3,7,8-TCDD used as reference compound. [c] 2,3,7,8-TCDF used as reference compound.

that generated by this laboratory, were judged to compromised by the presence of significant levels of or interfering substances. In fact, the presence amounts of superfluous substances in a number analytes prevented the determination of TCDD in 5 samples and apparently produced positive inter- 3 fortified samples, as indicated by quantitative which were significantly greater than the levels of

Quantitation Procedures. Quantitations of 2,3,7,8- 2,3,7-TCDF, and OCDD are made directly by com- the integrated responses of the native compounds of the isotopically enriched marker compounds. is made by analysis of known amounts of the marker compound and an authentic quantitative of the native material under those GC/MS condi- and in analysis of samples.

the first 2 years of use of this procedure, quanti- other PCDDs, PCDFs, and non-ortho PCBs were the external standard technique using mixtures of 12 compounds. Toward the latter half of 1982, quantitations of these compounds were performed using major isotopic marker compounds as internal standards for all congeners. Usually [37Cl]-OCDD and for quantitation of OCDD and OCDF, and [13C]- TCDD and [37Cl]-2,3,7,8-TCDF were used for quan- of all other PCDDs, PCDFs, and non-ortho PCBs. relative response factors for the various congener were determined by GC/MS analyses of mixtures of marker compounds and a series of 20 synthesized PCDFs, and non-ortho PCB isomers.

attempt was made to determine the suitability, in terms accuracy and precision, of quantitations of all congener using the internal standards (isotopic marker com- The experiment involved GC/MS-MIM and GC/EC replicates each) of a mixture of 17 native PCDDs PCDFs and the 5 isotopically enriched marker compounds. This mixture was subsequently subjected to the enrichment procedure (5 replicates) and analyzed again by GC/MS-MIM and by GC/EC. The mean and standard deviations of the integrated responses of all compounds relative to that of [13C]-2,3,7,8-TCDD were determined by GC/MS, and 2,3,7,8-TCDF was used as the internal standard in GC/EC analyses (Table V). The level of variation as measured by standard deviation for GC/MS quantitations using the internal standard was twice that determined for the GC/EC analyses. The data indicate that GC/MS quantitations using TCDD or TCDF as an internal standard were significantly more precise for tetrachloro through heptachloro congeners than for OCDD and OCDF. In contrast, no such disproportionate trends in precision were observed in the GC/EC analyses. The large variations associated with OCDD and OCDF are believed to be in part a consequence of GC/MS instrumental problems which were being experienced at the time and not necessarily characteristic of these types of analyses. Analyses of the mixture following application of the enrichment procedure show that the mean standard deviation is increased but comparable to instrumental variation. Nevertheless, the results indicate an acceptable level of precision for GC/MS quantitations of Cl_4 through Cl_7 congeners using a TCDD or TCDF as an internal standard in samples subjected to the enrichment procedure.

Determinations of PCDDs, PCDFs, and non-ortho PCBs were routinely carried out in the electron impact GC/MS mode. The GC/MS-EI technique, in contrast to negative ion chemical ionization analysis, exhibits comparable sensitivity for the broad range of congeners and permits identification and quantitation of all components in a single analysis. Negative ion chemical ionization GC/MS (GC/MS-NICI) has been observed in this laboratory and elsewhere (70) to exhibit a markedly enhanced sensitivity to PCDFs relative to PCDDs and, generally, to the higher relative to the lower chlorinated congeners of both groups. The ability to determine tetrachlorodioxins and tetrachlorobiphenyls in particular suffers

...NICI, and consequently this technique is un... ...complete determination of PCDDs, PCDFs, and ... at part-per-trillion levels. On the other hand, ... much less sensitive to background (especially ... and hydrocarbons) or cocontaminant substances ... yielded more easily interpretable data.

...y of Extraction. The implicit assumption in ... using the internal standards incorporated at the ... of the procedure is that the behavior of an iso... ...ched compound will be identical with that of the ... present in sample. This assumption is ...rsed for all enrichment processes except that ...ction of residues from the sample matrix. The ... of extractability of bioincorporated contami... ...biological samples or sorbed residues from soils ... products is particularly important in studies ... and PCDFs. Studies of the biochemistry of ... and related compounds in mammalian systems ...blished that these compounds exhibit high ... affinities for a hepatic cytosol protein; con... ...action of some PCDDs, PCDFs, and non-ortho ... biological samples may involve more than the ... of these residues from solution in fatty deposits. ... been reported of the efficiency of extract of ... PCDDs, PCDFs, or non-ortho PCBs. On the ... comparisons of the results of interlaboratory ... IV) involving a wide variety of extraction ... used for identical samples of fish containing ... 2,3,7,8-TCDD have provided a reasonable ... extractability of this substance from fish tissue. ... these studies suggest that the neutral column ...ployed in this procedure is essentially equivalent ... extractions involving complete digestion of the ...ncentrated aqueous base or acid. Such digestions ... denature and hydrolyze all proteins and to ...erate all intact TCDD residues. Referring to ...boratory no. 1 in the USFDA study employed ... concentrated HCl; in the H&WC/USFDA ...tory no. 3 employed digestion with KOH, and ... employed digestion with HCl. Assuming that ... is as strongly bound in these samples of fish ... other PCDD, PCDF, or non-ortho PCB, the ...tion procedure is expected to effectively recover ...dues of these compounds. The effectiveness of ...action could be species dependent and cannot ... to other animal systems without similar studies ... Our rationale for addition of the internal ... to the samples at the beginning of the extraction ... than before homogenization and mixing of the ...nium sulfate was that equilibration of the native ... the internal standards could not be easily at... ...latter step. Consequently, losses in the homo... ...and drying step are not included in the internal ...quantitation procedure.

...adsorptive interaction of PCDDs, PCDFs, and ... PCBs with carbonaceous materials has been studied ..., and studies of fly ash containing these com... ...demonstrated that exhaustive extraction pro... ...required (72). Consequently, a study was un... ...in this laboratory to determine the relative effi... ...two methods of extraction of these compounds from ... sediment samples (73). The neutral column ... procedure was compared with a procedure (72) ... demonstrated to be effective for the recovery ... fly ash. Although the results of the com... ...were highly variable and no unambiguous ... relative efficiencies of the two procedures could ... of the procedures was uniformly superior

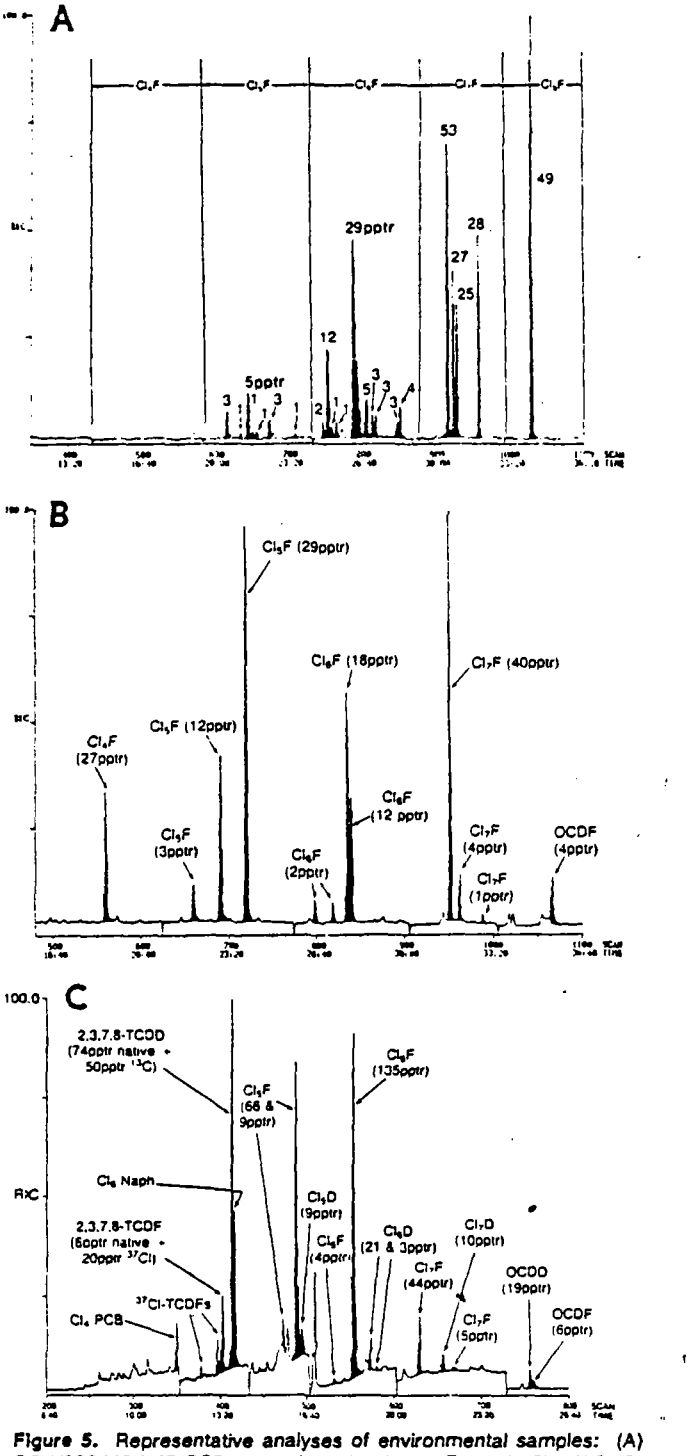


Figure 5. Representative analyses of environmental samples: (A) GC/NICI-MS-MID PCB contaminated soil from Fountain City, WI; (B) GC/NICI-MS-MID fish sample (carp) from Saginaw Bay at Bay City, MI; (C) GC/EI-MS-MID fish sample (carp) from the Niagara River at Ft. Niagara, NY.

to the other and appear to be roughly comparable in effectiveness. More definitive results are required from such studies before the efficacy of the column extraction procedure in analyses of soil and sediment samples can be established.

Applications to the Analyses of Environmental Samples. The procedure has been applied to the determination of PCDDs, PCDFs, and non-ortho PCBs in a wide range of sample types, primarily fresh-water fishes. The sample types which have been analyzed include about 12 species of fresh water fish (*55*, *68*) and three species of salt water fish (both whole body and fillet): snapping turtle fat (*54*), whole body

approximately five species of fresh water mussels, muscle and eggs of three species of birds, Baltic aquatic macroinvertebrates, commercial fish and terrestrial soils (*73*), soot from an office involving PCBs and polychlorinate benzenes (*26*), Aroclor 1260, and failed transformer fluid from site. The large majority of these samples were on the five Great Lakes and selected tributaries Mississippi, Hudson, and Sacramento Rivers, inboard rivers and estauries, and the Housatonic Massachusetts and Connecticut known to be contaminated with a wide range of persistent synthetic chemicals organochlorine pesticides, and industrial wastes. number of samples analyzed was approximately including over 50 control and procedural blank Essentially all of the 250 analyses were judged to according to the following criteria: (1) All marker were detected in the analyte. (2) An acceptable recovery (usually less than 5 pptr) was achieved. (3) and GC/MS properties of analyte components PCDDs, PCDFs, and non-ortho PCBs did not significant interferences. (4) The criteria for the identification of PCDDs, PCDFs, and non-ortho PCBs were

Representative multiple ion mass chromatograms of soil samples are presented in Figure 5. These GC/MS analyses of PCDDs, PCDFs, and non-ortho PCBs in three types of samples serve to exemplify the success of the procedure for such analyses. The GC/MS data is easily uncluttered by extraneous components, and interpretation of the data was routinely straightforward.

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...ctivity of Negative Ion Chemical Ionization Mass ...ctrometry for Benzo[*a*]pyrene

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...matography/negative ion chemical ionization mass ... (GC/NICIMS) was used as a selective and ... technique for the detection of benzo[*a*]pyrene (Ba... optimized conditions, the molecular anion, $M^{-\cdot}$, of ... more than 3 orders of magnitude more abundant ... its isomer benzo[*e*]pyrene (BeP) using methane ... gas. Quantities of BaP as low as 1 pg can ... detected in the selected ion monitoring mode and ... vs. concentration was linear over a range of 3 ... magnitude. The absolute sensitivity and the selec... detection were found to depend on the pressure and ... in the ion source of the mass spectrometer. ... was used for the quantitative determination of BaP, ...-cd]pyrene, and benzo[*ghi*]perylene in a sample ... crude oil as part of the process of certifying the ... Standard Reference Material.

... ion chemical ionization (NICI) mass spectra can ... from certain organic compounds by resonance ... of thermal electrons if the molecules have positive ... affinities, and if the internal energy of the molecular ... less than the electron affinity of the neutral species. ... major species formed is the molecular anion, $M^{-\cdot}$, ... yields relatively large ion currents and little ... The selectivity of NICI over electron impact ... well established and this feature has permitted ... wide applications over the past few years in the ... of compounds such as polychlorinated biphenyls (*1*), ...-3), pesticides (*1*, *4*, *5*), and nitrated polycyclic ... hydrocarbons (*6*). Ilda and Dashima (*7*) recently ... the methane negative ion chemical ionization mass ... of 21 polycyclic aromatic hydrocarbons (PAH). Oehme ... PAH in air particulate matter using NICI. He ... mixture of methane and nitrous oxide as the reagent ... promote ionization by electron capture and ion/mol... reactions and was able to differentiate isomeric PAH ... the relative abundances of various species formed. ... Capek, and Cooks (*9*) used negative ion chemical ... charge inversion mass spectrometry as a highly ... means for determining polycyclic aromatic hydro... in a solvent refined coal.

... used NICI mass spectrometry as a sensitive and ... technique for the quantitative determination of benzo[*a*]pyrene (BaP) in a sample of petroleum crude oil which is being certified as a Standard Reference Material (SRM). During the course of preliminary studies we have confirmed the large degree of selectivity for the detection of BaP over benzo[*e*]pyrene (BeP) noted by others (*7*, *8*). We have observed the molecular anion of BaP to be more than 1000 times more abundant than that of BeP under selected source conditions in the NICI mode using methane as the reagent gas. Our observations, reported here, show that the ion source pressure and temperature play an important role in the selectivity of detection for BaP. We have also observed excellent absolute sensitivity for the detection of BaP and are able to detect quantities as low as 1 pg in the selected ion monitoring mode.

EXPERIMENTAL SECTION

Negative ion chemical ionization mass spectra were recorded on a Hewlett-Packard 5985B quadrupole GC/MS system (Hewlett-Packard Co., Palo Alto, CA) with a dual EI/CI ion source and electronics capable of detecting negative ions. Chromatographic separations were carried out on a 30 m × 0.25 mm i.d. fused silica capillary column coated with a 0.25-μm film of a nonpolar liquid phase. Samples were injected in either the split or splitless modes with an injection port temperature of 300 °C and the column temperature was programmed from 200 to 300 °C at a rate of 4 °C/min. The column was interfaced directly to the ion source by inserting it through a 30 cm length of 0.16 cm o.d. stainless steel tubing. The stainless steel tubing also served as a conduit for introduction of the methane reagent gas (Matheson Ultra High Purity 99.97%) which was brought in coaxially with the capillary column. The pressure in the ion source was adjusted by varying the methane flow into the source via a flow controller. An ionization gauge, which was mounted approximately 15 cm from the source, was used to monitor the ion source manifold pressure. The pressure in the ion source itself was measured with a thermocouple gauge. Spectra were recored under conditions optimized empirically for the detection of BaP. The ion source was normally operated at 200 °C with a filament emission current of 300 μA and a primary electron beam energy of 60 eV. The mass spectrometer was calibrated in the NICI mode using ions at m/z 414, 452, and 633 from perfluorotributylamine and ions at m/z 233 and 235 from rhenium oxide generated by the filament. The ReO_3^- isotopes provide a good source of ions at low mass for tuning the mass spectrometer in the negative ion mode.

The PAH were obtained commercially: BaP (Community Bureau of Reference, BCR, Brussels, Belgium); BaP-d_{12} 98.6 atom % D (MSD Isotopes, St. Louis, MO); and BeP (Pfaltz and Bauer, Inc., Stamford, CT). The standards were analytical grade or higher and were used without further purification. Methylene chloride solutions of the PAH were prepared gravimetrically. The Wilmington crude oil sample was obtained from the Department of Energy and is one of the oils being stored in the EPA Repository

... address: Environmental Trace Substances Research ... University of Missouri, Columbia, MO 65201.

ATTACHMENT 3

PROCEDURES FOR FISH SAMPLE PREPERATION

SPECIFICS FOR THE CRAB ORCHARD PROGRAM

Only the edible portion of the fish will be analyzed for PCBs. The edible portion as defined by the FDA for the fish species collected from the Crab Orchard site are:

Carp	Fillet Skin On
Channel Cat	Fillet Skin Off
Bullhead	Fillet Skin Off
Largemouth Bass	Fillet Skin On

In addition, five fish of each species will be composited into one sample. Equal portions of the ground fillets will be mixed into one. The remaining portions will be stored frozen.

Prior to processing, each fish will be weighed and its length measured.

SAMPLES

This section gives general guides for preparing and compositing routine samples. It does not provide for handling the unusual sample. Because complete background information on samples is ordinarily unknown, and since residue analysts are usually unaware of what residues are present or of how they were incurred, *no sample should be assumed to be routine.*

A thorough visual examination of the gross sample should always be made before any preparation or compositing is begun. This should be on a sub by sub basis if sample is received in subsample form. A key to proper sample analysis can often be found by observation of the general appearance and odor of the product. Presence of soil, dust, wax, powder or stains; and foreign or off odors should be noted and recorded. When appearance or odor of the sample (or any of its subs) is unusual, the applicability of instructions in 141 and 142 should be carefully weighed before preparation and compositing are begun.

141

SAMPLE PREPARATION

Where samples are analyzed to determine whether they are in compliance with the Federal Food, Drug, and Cosmetic Act, they must be prepared for analysis according to preparation specified in the Regulations[1] or in Administrative guidelines which have been established for the residue on the commodity. The various ways to prepare raw agricultural and processed foods are given in 141.1 and 141.2 as an aid to residue analysts in proper choice of how to handle residue samples.

The portion of sample taken for analysis must be representative of the gross laboratory sample. It must be carefully handled to prevent loss of residue by volatilization and to prevent concentration of residue through physical separation of product during preparation. Meaningful residue data can only be obtained when integrity of sample is preserved. Haphazard preparation results in data that is useless and often misleading.

[1] Federal Food, Drug, and Cosmetic Act Regulations. Published in the Code of Federal Regulations, 40 CFR, part 180 - Protection of Environment, and 21 CFR, part 121, and 21 CFR, part 122 - Food and Drugs.

141.1 Raw Agricultural Commodities. Raw agricultural commodities include, among other things: fresh fruits, whether or not they have been washed, colored or otherwise treated in their unpeeled natural form; vegetables in their raw or natural state, whether or not they have been stripped of their outer leaves, waxed, prepared into fresh green salads, etc.; grains, nuts, eggs, raw milk, meats, and similar agricultural produce.
There are different ways required for preparing raw agricultural commodities for residue analysis. The various preparations are described as follows:

(1) Whole Raw Agricultural Commodity. — Most tolerances have been established on the product in its raw or natural state as shipped in interstate commerce. The whole raw agricultural product is prepared for analysis as in 141.12a.

(2) Whole Basis According to Regulation 40 CFR 180.1(j).—This regulation directs which portion of the commodity is to be discarded and which portion is to be taken for analysis, and is in accordance with how most tolerances were established on these products. Commodities for which preparation has been specified are listed in 141.12b, along with their regulation reference. This preparation is considered "whole basis" preparation for these commodities only.

(3) Whole Basis According to Specific Tolerance Regulations (40 CFR 180 Subpart C).—Special preparation for certain commodities is directed by the individual tolerance regulation. When samples are selectively collected for a specific residue, consult the tolerance regulation to determine if the portion of the commodity to be analyzed is specified.

(4) Edible Portion.--Inedible portions of the product are discarded and edible portion only is analyzed. The edible portion preparation for several commodities is listed in 141.12c. Analysts should use discretion in determining the inedible portion of products not listed in that section.

Analytical report must give full description of product as received for analysis and must clearly state the exact portion of food used for analysis.

141.11 Guide to Determining How to Prepare Raw Agricultural Commodities. The criteria for using the various preparation procedures for raw agricultural commodities (141.1) are listed here to aid the residue analyst in determining how to prepare samples for analysis. Determine, from any information available, the type of sample and reasons for analysis. Use the preparation procedure for that type sample.

Sample Type	Criteria	Procedure
I-R	Meets *ALL* of the following criteria: (a) Collected for multi residue determination. (b) No background information *or* background information does not indicate likelihood of contamination by specific pesticide or industrial chemical. (OBJECTIVE SAMPLES). (c) Commodity has at least one residue tolerance established and that residue is determined by methodology to be used.	Consult 141.12b and, if product is listed there, prepare as directed; if not listed, prepare the whole raw agricultural commodity as in 141.12a
II-R	Meets *EITHER* of the following criteria: (a) Selectively collected for a particular residue for which a tolerance is established. (b) Analysis of a type I-R sample reveals a significant residue which has an established tolerance on the product.	Consult the specific tolerance regulation listed in 40 CFR 180 Subpart C or 21 CFR 122 for preparation that may be required by regulation.
III-R	Meets ANY of the following criteria: (a) No tolerances have been established for any residue on the commodity or the chemicals with established tolerances on the commodity are not determined by methodology used. (b) Selectively collected for a particular residue for which no tolerance has been established on that particular commodity (e.g. endrin in melons). (c) Collected from an area where a known residue problem exists for a chemical for which no tolerance has been established on the particular commodity. (d) Analysis of a sample prepared as in 141.12a or in 141.12b ("whole basis") reveals significant quantity of a residue for which no tolerance has been established on the particular commodity.	Prepare sample according to the edible portion guide 141.12c.

141.12 Preparation of Raw Agricultural Commodities

141.12a Whole Raw Agricultural Commodity. Remove obviously decomposed leaves, berries, etc. Prepare the whole raw agricultural product. See 141.1(1).

141.12b Whole Raw Agricultural Commodity with Preparation Specified in 40 CFR 180.1(j). Prepare commodities listed in table below according to preparation in column b. See 141.1(2). Preparation given in Editors' Notes is in keeping with current policy.

141.12c Edible Portion. Prepare commodities listed in table below according to preparation in column c. See 141.1(4).

Commodity	Preparation	
	b Specified in 40 CFR 180.1(j)	c Edible portion
Bananas	Remove and discard crown tissue and stalk. 40 CFR 180.1(j)(1) (Editors' Note: Several specific tolerance regulations establish separate level for pesticide in pulp.)	Remove and discard peel; examine pulp only.
Corn, sweet	(Editors' Note: Some tolerance regulations specify portion for analysis as "kernels plus cob; husks removed.")	Remove and discard husks and cob; examine kernels.
Crabs, hard shell	(Editors' Note: Use edible portion guide.)	Examine a homogeneous mixture of meat and fatty materials isolated as described below: Heat crab in boiling water or place in autoclave under flowing steam for one minute if previously frozen, or five minutes if sample has been merely chilled and is possibly still alive. Remove claws and other appendages and pick out meat. Remove back shell. Clean out and discard viscera and gills (easily remove by hand).

Commodity	Preparation	
	b Specified in 40 CFR 180.1(j)	c Edible portion
		Include in the edible portion fatty material (yellowish colored) from inside tips of the back shell and any fatty material (yellowish colored) adhering to meat. Break crab in half and remove meat from body cavity excluding shell and other obviously extraneous materials.
Crab, soft shell	(Editors' Note: Use edible portion guide.)	Examine entire crab.
Eggs	(Editors' Note: Use edible portion guide.)	Discard shells; examine combined yolks and whites.
Fish (raw)	(Editors' Note: Use edible portion guide.)	Remove and discard heads, scales, tails, fins, guts and inedible bones; do not remove skin; fillet to obtain all flesh and skin from head to tail and from top of back to belly on both sides. Where extremely large whole fish are to be analyzed and filleting is impractical, 3 cross-sectional slices from each fish may be taken and combined.

Commodity	Preparation	
	[b] Specified in 40 CFR 180.1(j)	[c] Edible portion
		Clean, scale and eviscerate fish. Take 1" thick slices, one from behind the pectoral fins, one from half way between first slice and the vent, and one from behind the vent. Remove bones from each slice before combining.
		Rule of edibility supersedes these directions; e.g., catfish skin (inedible) is discarded.
Fruits (general comment)		Remove and discard stems.
Fruits, stone		Remove and discard stones or pits.
Garlic bulbs	Remove and discard roots, stems and outer sheaths (or husks); examine garlic cloves only. 40 CFR 180.1(j)(5)	Same as preparation in 40 CFR 180.1(j)(5)
Mangoes		Remove and discard rind and stone.
Melons	Remove and discard stems. 40 CFR 180.1(j)(4)	Remove and discard rind, stem and seeds; examine edible portion.

Commodity	Preparation	
	[b] Specified in 40 CFR 180.1(j)	[c] Edible portion
Nuts	Remove and discard shells. 40 CFR 180.1(j)(2)	Same as preparation in in 40 CFR 180.1(j)(2)
Oysters, Clams (raw)	(Editors Note: Use edible portion guide.)	Examine a homogeneous mixture of meats and liquor.
Peanuts	(Editors Note: Use edible portion guide.)	Remove and discard shells.
Pineapple	Remove and discard crowns (leaves at the top of the fruit). 40 CFR 180.1(j)(7)	Remove and discard crown and flowers (outer protective petals); examine edible portion only.
Pumpkins		Remove and discard rind, stem and seeds; examine edible portion only.
Root crops (general comment)	(Editors Note: Use edible portion guide.)	Rinse lightly to remove adhering soil.
Root vegetables including tops or with tops	Examine the roots and tops separately. Neither the pesticide residues on the roots nor on the tops shall exceed the tolerance level, except that in the case of carrots the tops shall be removed and discarded before analyzing roots for pesticide residues. 40 CFR 180.1(j)(6)	Same as preparation in 40 CFR 180.1(j)(6)
Shrimp (raw), crawfish and similar shellfish	(Editors Note; Use edible portion guide.)	Remove and discard heads, tails and shells; examine edible meat only.

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Commodity	Preparation	
	[b] Specified in 40 CFR 180.1(j)	[c] Edible portion
Strawberries	Remove and discard caps (hulls). 40 CFR 180.1(j)(3).	Same as preparation in 40 CFR 180.1(j)(3).

141.22 Preparation of Processed Foods

141.22a "As is" Product. Prepare the "as is" food (including concentrates, dehydrated foods, etc.) as recieved or as when introduced into interstate commerce.

141.22b Certain Commodities with Specified Preparation. Prepare as directed below:

Canned foods	Examine a homogeneous mixture of can contents; except, drain and discard brine and remove pits and stones.
Cheese	Do not remove or discard natural cheese rind. Do remove and discard waxed or oiled rings. Grind, dice, shred or blend cheese. See 142.22b.
Citrus pulp, Milk, Tomato pomace	Examine produce as received or as when introduced into interstate commerce.
Fish, breaded, raw or cooked	Do not remove breading. Fillet as necessary (as described in 141.12 "fish (raw)") to remove bones and/or tails.
Fish, canned in brine or water	Drain and discard liquid, examine remainder.
Fish, canned in oil, broth or sauce	Examine a homogeneous mixture of can contents.
Fish, frozen	Thaw, drain and discard drainings. Fillet - use entire piece. Whole fish - proceed as in 141.12 "fish (raw)."
Fish, smoked	Proceed as in 141.12 "fish(raw)."
Frog legs	Discard bones; examine edible meat only.
Oysters and Clams, canned or frozen	Examine a honmogeneous mixture of meats and liquor.
Shrimp and similar shellfish, breaded	Examine as received.
Shrimp and similar shellfish, canned in brine	Drain and discard brine; examine edible meat.
Shrimp and similar shellfish, frozen	Thaw, drain and discard drainings. Remove and discard heads, tails and shells; examine edible meat only.

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141.2 Processed Foods. Processed foods include foods that have been processed, fabricated, or manufactured by cooking, freezing, dehydrating or milling.

The various ways of preparing processed foods for analysis are as follows:

(1) "As is" Product. The food or feed as shipped in interstate commerce is prepared for analysis. Concentrates, dehydrated foods, etc. are analyzed "as is". Do not reconstitute to whole basis before analysis. See 143.12b for reporting results on concentrates and dehydrated products. Prepare low fat dairy products (e.g., skim milk, buttermilk, nonfat dried milk and uncreamed cottage cheese) on an "as is" basis. See 143.12a for reporting results on low fat dairy products.

(2) Specific Product Preparation - Special preparation is specified for certain processed foods in 141.22b.

141.21 Guide to Determining How to Prepare Processed Foods. Determine the type of sample and reasons for analysis. Use the preparation procedure for that type sample.

Sample Type	Criteria	Procedure
I-P	Meets ALL the following criteria: (a) Collected for multi residue determination (b) No background information or background information does not indicate likelihood of contamination by specific pesticide or industrial chemical.	Consult 141.22b and if product is listed there, prepare as directed; if not listed, prepare the "as is" product as received or as introduced into into interstate commerce.
II-P	Meets ANY of the following criteria: (a) Selectively collected for particular residue(s) (b) Selectively collected because of suspected likelihood of particular residue. (c) Collected from an area where a known residue problem exists. (d) Analysis of a type I-P sample reveals a significant residue for which a tolerance is established on the product analyzed.	Consult the specific tolerance regulations listed in 21 CFR 121 or 21 CFR 122 for preparation that may be required by regulation. If no tolerance is established for the residue in the particular processed food, the analyst must decide, based on the particular circumstances, how best to prepare sample.

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142

SAMPLE COMPOSITING

Composite prepared sample according to guidelines in 142.2 by chopping, grinding, blending, etc. to obtain homogeneous mixture. The relatively small portion (25-100 g) of prepared composite that is taken for analysis must be representative of gross laboratory sample. Routine chopping, grinding, blending, etc. does not always produce a proper homogenate, as is the case with dried hays and some fish samples. When product is not visibly homogeneous, use standard mixing and quartering techniques to insure that portion for analysis is representative. See 142.4 for notes on preparing composites for analysis. Select representative portion of uniformly mixed sample for analysis.

142.1 Portion of Sample for Dithiocarbamate Analysis. Some dithiocarbamate compounds decompose rapidly in presence of slurry of crop material. Cullen (Anal. Chem, 36, 221-224 (1964)) reported that speed is essential as soon as surface of crop is broken and dithiocarbamate is in intimate contact with water, enzymes, and sugars. He noted a rapid decrease in recovery with time of contact in aqueous crop solution and recommended that samples for dithiocarbamate analysis be either analyzed immediately after harvest or frozen for storage.

When dithiocarbamate residues are to be determined, select representative units for dithiocarbamate analysis prior to chopping, grinding or blending sample. Where sample units are small and free flowing (e.g. grains, beans, berries, etc.), mix well and take whole units for analysis; where sample units are large, take wedges from each unit. Analyze immediately or freeze immediately after cutting. An exception to above is where commodity contains free juices (e.g. tomatoes, apples, oranges, etc.) and requires cutting in pieces to fit into apparatus. In such cases, take representative whole units and freeze before cutting. Dice frozen units without allowing them to thaw; mix and take sample for analysis.

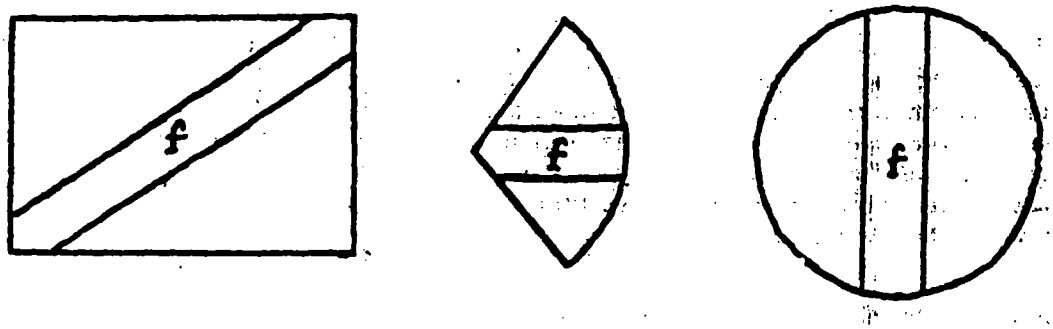
142.2 Guidelines for Preparation of Composites.

142.21 Total Sample Composited and Comminuted. Where practical, comminute and thoroughly mix entire prepared sample. See 142.1 for portion to be removed if sample is to be analyzed for dithiocarbamates.

142.22 Total Sample Composited and Fraction of Sample Comminuted.

142.22a Product with Small Units. Where sample product consists of small units (e.g., grains, cherries, nuts, dried peas and beans) and it is not practical to prepare and comminute entire sample, mix and quarter down to approximately 4 pounds or 4 quarts. From quartered sample, prepare product as in 141.1 or 141.2 and chop or grind prepared sample to obtain minimum of 1 pound or 1 quart comminuted sample for original analysis. See 142.4(4) for grinding low moisture products.

142.22b Product of Homogeneous Nature. Where large sample of homogeneous nature must be composited (e.g., butter, cheese), and melting entire sample of butter or dicing, shredding, or blending entire sample of cheese is not practical, prepare sample for original analysis by taking equal portions from each packaged unit. Where large blocks, wedges or wheels of cheese are to be prepared, take fraction (f) for analysis as in diagram below.



Prepare cheese as in 141.22b(2) and composite by dicing, shredding or blending.

142.23 Composite of Individual Subdivisions. Where identity of subdivisions must be maintained for possible additional analysis of individual subs, prepare composite as follows:

(1) Animal tissue	Grind each sub (meat grinder). Composite 100 g from each sub and grind again.
(2) Dairy products	Equal weight from each sub. Grind, dice or blend.
(3) Eggs	Half of eggs in each sub. Blend. See 142.4(2).
(4) Feed, forage and hay	Quarter each sub down to 200 g (100 g for processed feeds and silage). Composite 200 (100) g from each sub. Chop or grind to pass 20 mesh. See 142.4(4).
(5) Fruits	(a) Large (apples, pears, tomatoes, etc.): sample each unit within sub. Composite an equal weight from each sub. Chop or blend.
	(b) Small: 200 g from each sub. Chop or blend.
(6) Grains	100 g from each sub after thorough mixing. Grind composite to pass 20 mesh. See 142.4(4).
(7) Milk	100 g (ml) from each sub after thorough mixing.
(8) Nuts	Remove and discard shells. Composite equal weight, 100 g or more, of nut meats from each sub. Chop or grind.
(9) Pod Vegetables	(Beans, peas, etc., also asparagus) 200 g from each sub after thorough mixing. Chop or grind.

(10)	Root Vegetables	Sample each unit within sub taking equal weight from each sub. Chop or grind.
(11)	Seeds	100 g from each sub after thorough mixing. Grind composite to pass 20 mesh. See 142.4(4).
(12)	Spices	200 g from each sub after thorough mixing. Grind or chop.
(13)	Stalk Vegetables	(Celery, broccoli, etc.). Quarter each stalk in sub lengthwise. Take two opposite quarters from each stalk and composite these quarters by chopping.
(14)	Vegetables	(a) Head: quarter each head in sub. Take two opposite quarters from each head and composite these quarters by chopping.

(b) Leafy

(1) Leaf cut: mix sub well and select leaves at random until 200 g portion is obtained. Composite 200 g from each sub and chop entire composite.

(2) Field cut (leaves attached to stalk): select bunches at random until 500 g portion is obtained. Composite 500 g from each sub and chop entire composite.

142.3 Portions of sample retained. Select three portions from total sample homogenate (142.21) and identify one as "original analysis", second as "check analysis", and third as "reserve" (for claimant.) Where fraction sample composites (142.22) and individual sub composites (142.23) have been prepared, retain prepared composite and reserve of sample. Seal and store all retained portions of sample in such manner as to prevent decomposition of product and residue. This requires that all products be frozen until findings of original analysis have been verified. The amount of composite retained is governed by extent of analysis required on sample. However, in no case should portions be less than one quart each (or for products of high density, one pound) for original, check, and reserve. Sample size for analysis is given in method.

142.4 Notes on compositing and comminuting.

(1) Using Hobart vertical cutter mixer. The 40 quart Hobart vertical cutter mixer was tested to determine its mixing and chopping ability. Varying quantities of several agricultural products were chopped for varying time intervals and resulting mixtures were checked for distribution and particle size. Based on this study, a minimum of 20 lbs. of compactly formed products (such as potatoes, beets, carrots, etc.) or a minimum of 1/2 bushel of loosely formed products (such as cabbage, lettuce, greens, etc.) is recommended for chopping composite in 40 quart Hobart vertical cutter mixer. Chop a minimum of five (5) min, stopping chopper and hand scraping material back into bottom

of chopper at least once during operation. (More, C.A., private communication, Food and Drug Adm., June, 1966).

(2) Blending eggs. Blend at low speed for minimum of five (5) min or until sample is homogeneous. Low speed blending will minimize foaming or "whipping" of sample.

(3) Thawing frozen composites. Thaw frozen comminutes completely and remix before portion is taken for analysis. Any liquid phase separation that occurs in freezing or thawing must be reincorporated into composite before taking sample for analysis.

* (4) Grinding low moisture products, oilseeds, and other difficult samples. Grind samples to fine mesh (ca 20 mesh) in Ultra Centrifugal Mill (see 111) or equiv. Grind oil seeds first through a large sieve (3-5 mm), then regrind through a fine (≤0.5 mm) sieve to minimize drag on the motor. Collect ground material in the 500-800 g capacity collecting pan and thoroughly mix several batches as necessary to provide appropriate sample size from which to take the analytical sample. (Sawyer, L.D., private communication, Food and Drug Admin., Jan., 1977.)

In the absence of a centrifugal mill, grind samples through a Wiley mill or equiv., taking care to prevent physical separation of the product in the mill. A stepwise grinding procedure, in which sample is coarsely ground, then quartered down and a smaller portion ground to 20 mesh or smaller, may be necessary with some products. Loss of volatile pesticides can occur during grinding where heat is generated in process. Dry Ice has been used to precool mills before sample is ground.

It may also be advisable to grind materials such as hay through the Wiley mill prior to final grinding through the centrifugal mill.

* (5) Grinding of fish. To prevent the skin of fish from clogging the grinder during the preparation of fish samples, the fish may be frozen prior to grinding. Sample handling must be consistent with the directions given in 141.12c and 141.22b, in terms of the portion of the sample retained for analysis. A distinction must be made between (1) fish frozen by a processor for sale as frozen fish and (2) raw fish sampled by an inspector and frozen for preservation prior to analysis. In the former situation, the sample must be thawed and the drainings discarded, no matter what further handling is required for analytical sample preparation. In the latter case, no drainings should be discarded and fish need be thawed only enough to facilitate preparation of the analytical samples.

Prepare raw fish (or fish sampled raw and frozen by the inspector) as described in 141.12c, then freeze in portions of suitable size for introduction into the grinder.

Thaw frozen fillets and discard drainings as described in 141.22b. Then refreeze in portions of suitable size for introduction into the grinder.

of chopper at least once during operation. (More, C.A., private communication, Food and Drug Adm., June, 1966).

(2) Blending eggs. Blend at low speed for minimum of five (5) min or until sample is homogeneous. Low speed blending will minimize foaming or "whipping" of sample.

(3) Thawing frozen composites. Thaw frozen comminutes completely and remix before portion is taken for analysis. Any liquid phase separation that occurs in freezing or thawing must be reincorporated into composite before taking sample for analysis.

* (4) Grinding low moisture products, oilseeds, and other difficult samples. Grind samples to fine mesh (ca 20 mesh) in Ultra Centrifugal Mill (see 111) or equiv. Grind oil seeds first through a large sieve (3-5 mm), then regrind through a fine (≤0.5 mm) sieve to minimize drag on the motor. Collect ground material in the 500-800 g capacity collecting pan and thoroughly mix several batches as necessary to provide appropriate sample size from which to take the analytical sample. (Sawyer, L.D., private communication, Food and Drug Admin., Jan., 1977.)

In the absence of a centrifugal mill, grind samples through a Wiley mill or equiv., taking care to prevent physical separation of the product in the mill. A stepwise grinding procedure, in which sample is coarsely ground, then quartered down and a smaller portion ground to 20 mesh or smaller, may be necessary with some products. Loss of volatile pesticides can occur during grinding where heat is generated in process. Dry Ice has been used to precool mills before sample is ground.

It may also be advisable to grind materials such as hay through the Wiley mill prior to final grinding through the centrifugal mill.

* (5) Grinding of fish. To prevent the skin of fish from clogging the grinder during the preparation of fish samples, the fish may be frozen prior to grinding. Sample handling must be consistent with the directions given in 141.12c and 141.22b, in terms of the portion of the sample retained for analysis. A distinction must be made between (1) fish frozen by a processor for sale as frozen fish and (2) raw fish sampled by an inspector and frozen for preservation prior to analysis. In the former situation, the sample must be thawed and the drainings discarded, no matter what further handling is required for analytical sample preparation. In the latter case, no drainings should be discarded and fish need be thawed only enough to facilitate preparation of the analytical samples.

Prepare raw fish (or fish sampled raw and frozen by the inspector) as described in 141.12c, then freeze in portions of suitable size for introduction into the grinder.

Thaw frozen fillets and discard drainings as described in 141.22b. Then refreeze in portions of suitable size for introduction into the grinder.

Thaw whole fish frozen for sale as frozen fish and discard drainings, then prepare as described in 141.22b. Refreeze in portions of suitable size for introduction into the grinder. Grind immediately three times in a Hobart Food Cutter (or equiv.) with grinder attachment. (Thompson, T.D., private communication, Food and Drug Admin., Feb., 1976).

Thaw whole fish frozen for sale as frozen fish and discard drainings, then prepare as described in 141.22b. Refreeze in portions of suitable size for introduction into the grinder. Grind immediately three times in a Hobart Food Cutter (or equiv.) with grinder attachment. (Thompson, T.D., private communication, Food and Drug Admin., Feb., 1976).

ATTACHMENT 4

SAMPLING EQUIPMENT

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

SAMPLING EQUIPMENT

PHASE I & II SAMPLING

HIT LIST (sub.1)	NUMBER or AMOUNT per UNIT (person, site, or sample)	X	UNITS	=	NUMBER OF TOTAL NO. NEEDED	WHERE AVAILABLE (here, St.Louis, etc.)
SAMPLING EQUIPMENT:						
SITE IDENTIFICATION:						
2"x2"x24" wooden stakes	100		-		100	
orange spray paint	2 cans		-		2	
rope	400 ft.		-		400 ft	
hammer	1		-		1	
WATER:						
waders	2size10; 2size12				4	
boat	1		-		1	
disposable stirrers						
2" submersible pump						
water filtering device						
filters						
100 ft. steel tape						
shovel						
electrical cord						
glass funnel						
deep buckets						
paper towels						
plastic sheeting	10'x50'roll		2		2 rolls	
polypropylene rope						
silicone spray						
flagging tape						
electrical tape						
aluminum foil	3 rolls				3 rolls	
tool kit: phillipshead screwdrivers	3 sizes				3	
wrenches	2 cresent; 2 pipe				4	
hammers	(1)5#; (1)1# claw				2	
knife	1				1	
pliers	1 ndle-nose; 1 reg.				2.	
tubing bender	(1) 3/8"				1	
tubing cutter	1				1	
saw	1				1	
? automatic cord reeler and lead cord						
SEDIMENT:						
aluminum pans	1 dozen					
Wildco hand operated core sampler						
2"OD, 1/16" thickness Lexan tubing	8 foot lengths		25		200 ft	
polypropylene scoops	6					
aluminum scoops	6					
disposable spatulas						
GEOPHYSICAL:						
pH meters						
specific conductance meters						
thermometers						
magnetometer/electromagnetic induction equipment						
surveying equipment						
water level probe	1				1	
[sampling trier						
[soil auger						
[split barrel sampler						
HANDLING, STORAGE, AND SHIPPING:						

CRAB ORCHARD NATIONAL WILDLIFE REFUGE
SAMPLING EQUIPMENT

PHASE I & II SAMPLING

HIT LIST (sub.1)	NUMBER or AMOUNT per UNIT (person, site, or sample)	X UNITS	= TOTAL NO. NEEDED	WHERE AVAILABLE (here, St.Louis, etc.)
coolers	(10) 20 quart		10	
sampler jars (see BOTTLES file)				
freezer	1		1	
van with roofrack	1		1	
dry ice				
ice				
shipping labels				
markers				
DOCUMENTATION:				
field notebooks				
sample tags				
record sheets				
chain-of-custody records				
camera	1		1	
35 mm, color slide film				
QA/QC:				
field blanks				
duplicate samples				
split samples				
ignitability field test				
SAFETY EQUIPMENT:				
GENERAL PERSONAL GEAR (SAFETY LEVELS B, C, AND D):				
calibrated HNU-1101 photoionizing air monitir				
pressure demand self-contained breathing apparatus; high effeciency organic vapor/particulate/pesticide cartridge (respiratory safety level C)				
rubber safety boots or safety workboots with rubber overboots (safety levels B, C, & D)				
cotton overalls (level D) or work clothing under white tyvek suit (levels B & C)				
tyvek or other hood (levles B & C)				
cotton gloves (level D) or surgeon's gloves with rubber overgloves (levels B & C)				
protective eyewear				
hard hat (during drilling)				
noise protection (during drilling)				
2 way communication				
chemical resistant clothing(yellow tyveks,PVCcoveralls, or butyl apron) as needed for specific tasks				
first aid kit and manual				
LEVEL A-HIGHEST LEVEL OF RESPIRATORY,SKIN,AND EYE PROTECTION:				
THE ABOVE LIST PLUS:*				
chemical-resistant fully encapsulating suit				
chemical-resistant outer and inner gloves				
chemical-resistant boots with steel toe and shank.. disposable protective suit, gloves, and boots (worn over fully encapsulating suit)				
LEVEL B:				
THE GENERAL GEAR LIST PLUS:*				
chemical-resistant clothing (overalls and long-				

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

SAMPLING EQUIPMENT

PHASE I & II SAMPLING

HIT LIST (sub.1)	NUMBER or AMOUNT per UNIT (person, site, or sample) X	UNITS = NEEDED	TOTAL NO.	WHERE AVAILABLE (here, St.Louis, etc.)
sleeved jacket; coveralls; hooded, one or two-piece chemical-resistant splash suit; disposable chemical-resistant coveralls).....				
chemical-resistant outer and inner gloves.....				
chemical-resistant boots with steel toe and shank..				
chemical-resistant disposable outer boots.....				
hard-hat with face shield.....				
LEVEL C:				
THE GENERAL GEAR LIST PLUS:*				
chemical-resistant clothing (coveralls; hooded,two-piece chemical-resistant spalsh suit; chemical-resistant hood and apron; disposable, chemical-resistant coveralls).....				
chemical-resistant outer and inner gloves.....				
chemical-resistant boots with steel toe and shank..				
chemical-resistant disposable outer boots.....				
hard-hat with face shield.....				
escape mask.....				
LEVEL D:				
THE GENERAL GEAR LIST PLUS:*				
boots/shoes:leather or chemical-resistant with steel toe and shank.....				
chemical-resistant disposable outer boots.....				
hard hat with face shield.....				
escape mask.....				
safety glasses or chemical splash goggles.....				
DECONTAMINATION:				
brushes.....	6		6	
tub.....				
acetone.....	1 gal		1 gal	
hexane.....	1 gal		1 gal	
distilled water.....	5 gal		5 gal	
55 gallon drums.....	4		4	
interference-free, redistiiled solvent (eg. acetone or methyl chloride).....				
detergent.....				
5 gallon pails.....				
kiddie pool.....	2		2	
Gateraid.....	2 cases		2 cases	
fire extinguishers.....	1 ABC		1	
Jerry jugs.....	(4) 5 gal		4	
plant sprayer.....	1 10 gal		1	
air horns.....	2		2	
plastic garbage cans.....	2		2	
scissors.....	2 *pair*		2 *pair*	
pocket knives.....	3		3	
Janitor-in-a-drum cleaner.....	1		1	

*(MAY INCLUDE MORE SPECIFIC DESCRIPTIONS OF SOME GENERAL LIST ITEMS)

NOTE:Level B protection should be available in the event that the nature and hazards of a site are unknown, and must be further defined by on-site studies. Safety levels can later be modified appropriately.

aamount	SYRACUSE	check	amount	St.LOUIS	check	amount	REFUGE/MARION	check
	LAB			VAN WITH ROOFRACK			BOAT (REFUGE)	
	---						ICE (MARION)	
	SAMPLING JARS (see pages 2&3)			DRY ICE			DISTILLED WATER	
	RECORD SHEETS			COOLERS			(EITHER AT AN INDUSTRY	
	CHAIN-OF-CUSTODY RECORDS			ALUMINUM PANS			OR FROM SOUTHERN	
	DETERGENT			PLASTIC PAILS			ILLINOIS UNIVERSITY)	
	SOLVENT (NANOGRADE):			WADERS			FREEZER	
	ACETONE			TOILET BRUSHES			STATION WAGON	
	HEXANE			KIDS POOL				
	WATER FILTERING DEVICE			GATERAID				
	FILTERS			FIRE EXTINGUISHER				
	DETERGENT			JERRY JUGS				
	---			PLANT SPRAYER				
	HYDROGEOLOGIC			PLASTIC SHEETING				
	---			AIR HORNS				
	WATER LEVEL PROBE			GARBAGE CANS				
	ELECTROMAGNETIC EQUIP'T			SCISSORS				
	SURVEY EQUIPMENT			POCKET KNIVES				
	---			JANITOR-IN-A-DRUM				
	SUPPLY			ALUMINUM SCOOPS				
	---			POLYPROPYLENE SCOOPS				
	SHIPPING LABELS			LEXAN TUBING				
	MARKERS			ROPE				
	I.D.STICKERS(RED&YELLOW)			HAMMER				
	RUBBERBANDS			STAKES				
	FOLDERS			ORANGE PAINT				
	---			ALUMINUM FOIL				
	DIV. 3			HACKSAW				
	---			100 ft. STEEL TAPE				
	CAMERA			SHOVEL				
	COMPUTER SAMPLE LABELS			ELECTRICAL CORD				
	pH METER			GLASS FUNNEL				
	SPEC. CONDUCTANCE METER			DEEP BUCKETS				
	SAFETY EQUIPMENT (see page 4)			PAPER TOWELS				
				POLYPROPYLENE ROPE				
				SILICONE SPRAY				
				FLAGGING TAPE				
				ELECTRICAL TAPE				
				DISPOSABLE SPATULAS				
				DISPOSABLE STIRRERS				
				THERMOMETERS				
				DRUMS				
				SQUEEZE BOTTLES				
				TUB				
				INTERFERENCE-FREE				
				REDISTILLED SOLVENT				

ATTACHMENT 5

GENERAL PROCEDURES FOR PCB'S

PROCEDURES FOR ORGANOCHLORINE PESTICIDES AND PCBs IN ENVIRONMENTAL MATRICES REVISED AUGUST 1986

1.0 INTRODUCTION

1.1 The following procedures are for organochlorine pesticides and PCBs in environmental matrices. The procedures are applicable to the pesticides and PCBs listed in Table 1. The procedures are gas chromatographic (GC) methods written for use at the laboratory bench level. As such, a space is provided on the right hand side for notes and comments.

Note: See Table 10, pgs. 5, 6, 18 and 19 for detection levels and QA/QC requirements.

2.0 METHODOLOGY

2.1 The procedures are adopted from methods developed by EPA and State organizations. The principal source of reference is the EPA Contract Laboratory Protocol (CLP) which has also been adopted by NYS DEC for its Superfund programs.

2.2 It is not the intent of this document to rewrite the procedures developed by EPA with the same degree of detail. Instead, our purpose is to adopt the EPA procedures to our operation. As such, it is important that the analyst reads and becomes familiar with the EPA procedures as well as this document. The referenced procedures are copied and attached for your review.

2.3 Other referenced materials include the EPA Method 608 which is designed for industrial and municipal discharges. Also, a copy of the "Guidelines on Analytical Methodology for Pesticide Residue Monitoring" is attached for your review. This document provides a broader perspective of pesticide analysis.

2.4 Incorporated into this document by reference is the safety section of The 16th Edition of Standard Methods, Section 108. This is mandatory reading in the laboratory.

3.0 NOTES ON THE PROCEDURES

3.1 All glassware must be specially cleaned for trace organics work. The washing procedure is outlined in detail in Appendix 1.

7.0 FLORISIL COLUMN CLEAN-UP (MACRO COLUMN)

7.1 Florisil column chromatography effectively removes interfering organics from the sample extract and roughly separates the pesticides into polar and nonpolar fractions. The pesticide separation profiles achieved by this technique are presented in Figures 2-1 through 2-4. The analyst must demonstrate this same degree of separation for each new batch of florisil used. The procedure for its verification is to run 1ml of a 20ppm mixture of pesticides through a column packed with the new lot of florisil. The eluates are collected in 50ml fractions and directly analyzed on the GC, however, the eluates of 100% methylene chloride must be solvent switched to hexane first.

7.2 To prepare the column, place a small glass wool plug at the base of an 11 X 500mm glass chromatographic column. Dry pack the column with 10g of 60/120 mesh florisil, PR grade, and another inch of anhydrous sodium sulfate. Tap column lightly until evenly packed.

7.3 Pre-elute column and wash with 50ml of hexane. Prior to the exposure of sodium sulfate layer to air, add 1-5ml of the sample extract to the column. Again, just prior to the exposure of the sodium sulfate layer to air, add 100ml of 10% methylene chloride in hexane. Collect eluate, fraction 1, into a 250ml Kuderna-Danish evaporator. Next add 150ml of 100% methylene chloride and collect eluate, fraction 2, into a 250ml Kuderna-Danish evaporator.

7.4 Concentrate both fractions to approximately 2ml in a water bath. Fraction 2 must be solvent switched to hexane by adding 50ml of hexane when the methylene chloride is approximately 2ml, then reconcentrate to desired level.

7.5 Adjust extracts to final volume (usually 1ml) by dilution with hexane or evaporation with a gently stream of purified nitrogen.

7.6 Sulfur interferences usually elute in the first fraction. Add copper metal to extract if sulfur is suspect.

8.0 FLORISIL CHROMATOGRAPHY (MACRO COLUMN)

8.1 Place a glass wool plug in the bottom of a disposable glass Pasteur pipet (1cm O.D. X 5 3/4" long). Add 1.5 grams of florisil and tap gently. Add 0.5 grams of sodium sulfate leaving at least an inch of space in the top of the pipet.

8.2 Pre-elute the column with 10ml of hexane and discard. Just prior to the exposure of the sodium sulfate to air, add 1-2ml of the extract to the column. Again, just prior to the exposure of the sodium sulfate to air, add 10ml of hexane in incremental quantitites.

8.3 Concentrate the cleaned extract to 1ml of the nitrogen blowdown apparatus.

8.4 This cleanup procedure effectively separates the nonpolar PCBs from polar organics. However, it also removes the DBC surrogate from the extract.

9.0 ALUMINA COLUMN CLEANUP

9.1 Add 3gm of Activity III neutral alumina to the 10ml chromatographic column (K-420160). Tap to settle the alumina.

9.2 Transfer 1ml of the extract (which has been adjusted to 50:50 acetone and hexane) to the column. Elute the column with 9ml hexane.

9.3 Concentrate to 1ml on the nitrogen blowdown apparatus.

9.4 Add copper metal to the extract if sulfur is suspected.

10.0 SULFURIC ACID CLEANUP (PCB ONLY)

10.1 Transfer 5ml of the extract to a 12ml glass vial with a Teflon liner. The extract must be hexane or iso-octane for the procedure to work.

10.2 Add 5ml of concentrated sulfuric acid to the vial.

10.3 Vortex for 1 minute, then allow the phases to separate.

10.4 Vial 1ml of the organic extract for analysis.

10.5 This procedure will destroy the DBC surrogate if present.

11.0 GAS CHROMATOGRAPHY

11.1 The proper operation of a gas chromatograph requires several years of experience. An analyst becomes proficient by learning from his colleagues, by reading literature and by attending training seminars. Some of the fundamentals of GC analyses are described in the following sections. For the specifics, consult the EPA CLP Protocol.

11.2 Samples processed for pesticides and PCBs are analyzed on a mixed phase polar column. Samples requiring only PCBs are analyzed on a nonpolar column. The instrument conditions are as follows:

Column: 1.5% SP2250 + 1.95% SP2401 on 100/120 Supelcoport packed in a 6ft. X 4mm ID glass column
Oven Temperature: 160°C to 200°C at 20° per minute, 1 minute initial hold

Column: 3% OV-1 on 80/100 Supelcoport packed in a 6ft. X 2mm ID glass column
Oven Temperature: 160°C to 200°C at 10° per minute, 1 minute initial hold

11.3 The presence of a pesticide or PCB in a sample is determined by the retention time of the compound(s) on the GC column compared to the retention time of the standard(s) run under identical conditions. It is, therefore, imperative that the retention times of the standard and samples be reproducable throughout the run period. This is verified in two ways. DBC is added to every sample and to the standards so the retention time of DBC is used as an index. The second method of verification is to run standards in the beginning, in the middle and at the end of the run period.

11.4 If a pesticide is detected in a sample, its presence must be confirmed by analysis on a different column or by GC/MS techniques. In most case, the primary column is the 1.5% SP2250 + 1.95% 2401, a polar column. The confirmation column is the 3% OV-1, a nonpolar column. Standards and the sample are analyzed consecutively for confirmation.

11.5 For every batch analysis the linearity of the instrument must be defined by running three standards at three concentration levels. The response factors from these three runs must be within 10% of each other. If a pesticide or PCB is detected in a sample, it must be diluted into the linear range before it is quantitated.

12.0 REFERENCES

a. USEPA Contract Laboratory Program, July 1985 Revision

b. EPA Method 608, Federal Register, 40CFR, Part 136, October 26, 1984

c. Standard Methods for the Examination of Water and Wastewater, 16th Edition, APHA, AWWA, WPCF, 1985

d. Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue, EPA 600/4-81-055

e. Food and Drug Administrations Pesticide Analytical Manual, Volume 1, March 1, 1975

f. Guidelines on Analytical Methodology for Pesticide Residue Monitoring, Federal Working Group on Pest Management, Washington, D.C., June 1975

g. Organochlorine Pesticides and PCBs in Fish Tissue Samples, Michigan Department of Natural Resources, Received June 28, 1985

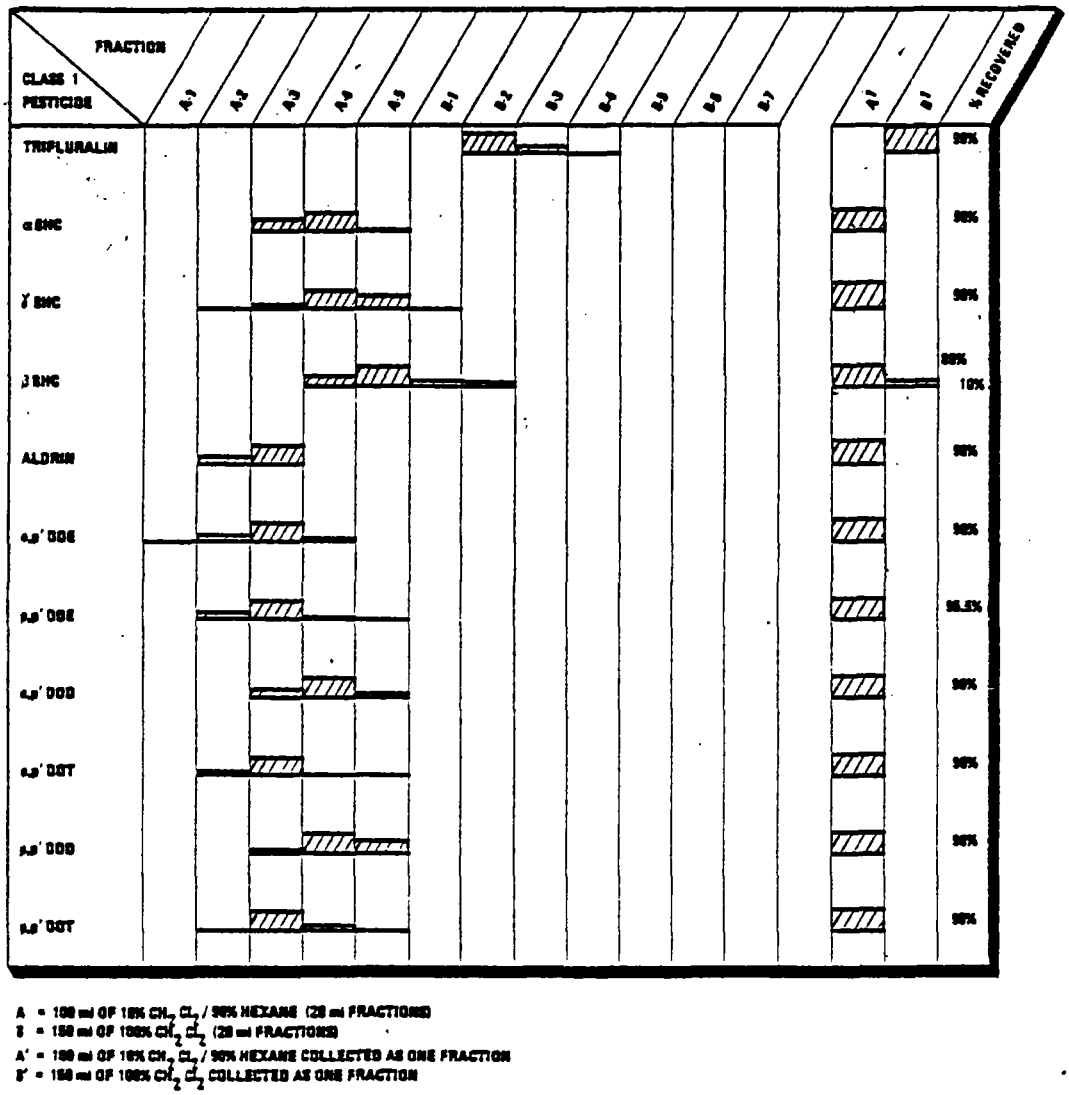
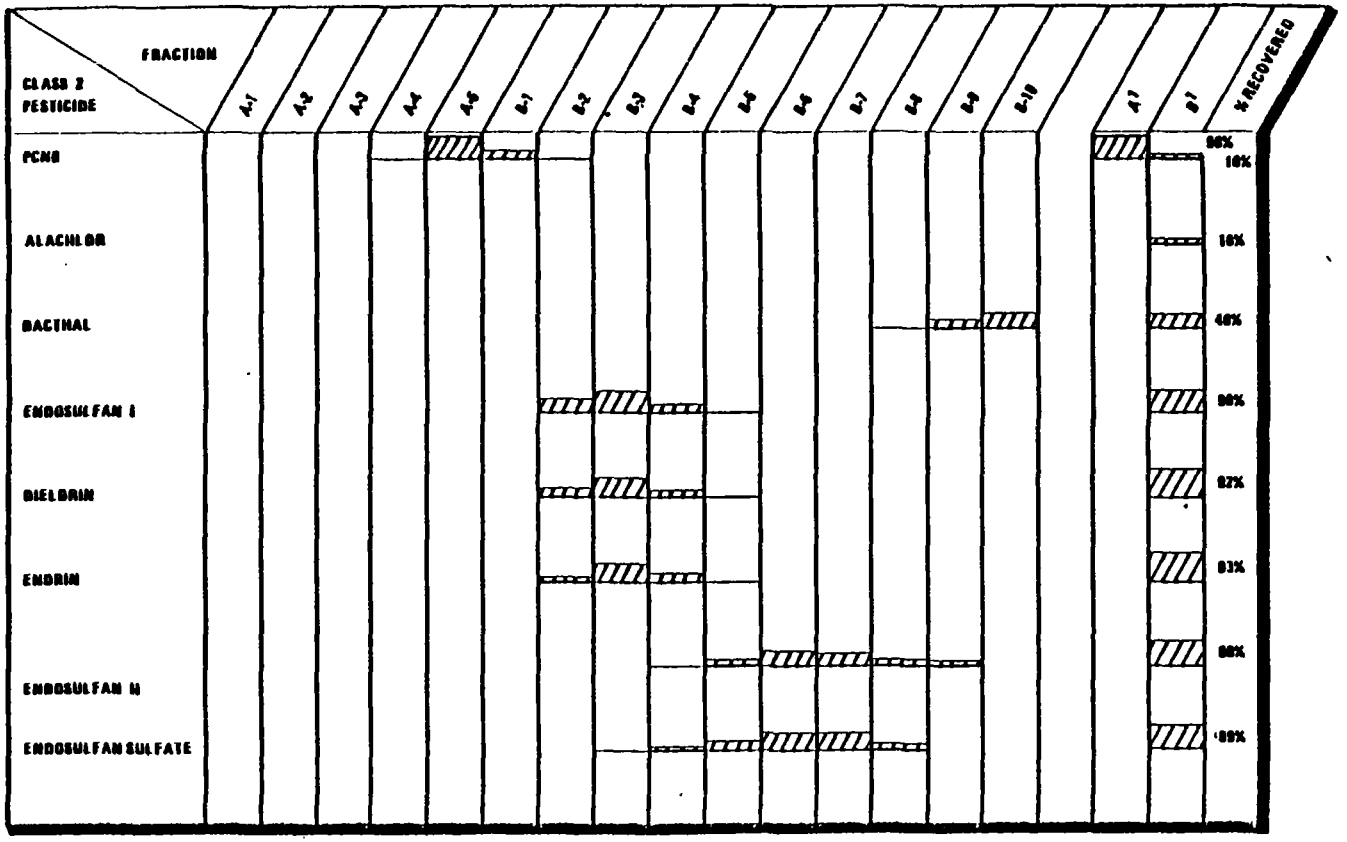
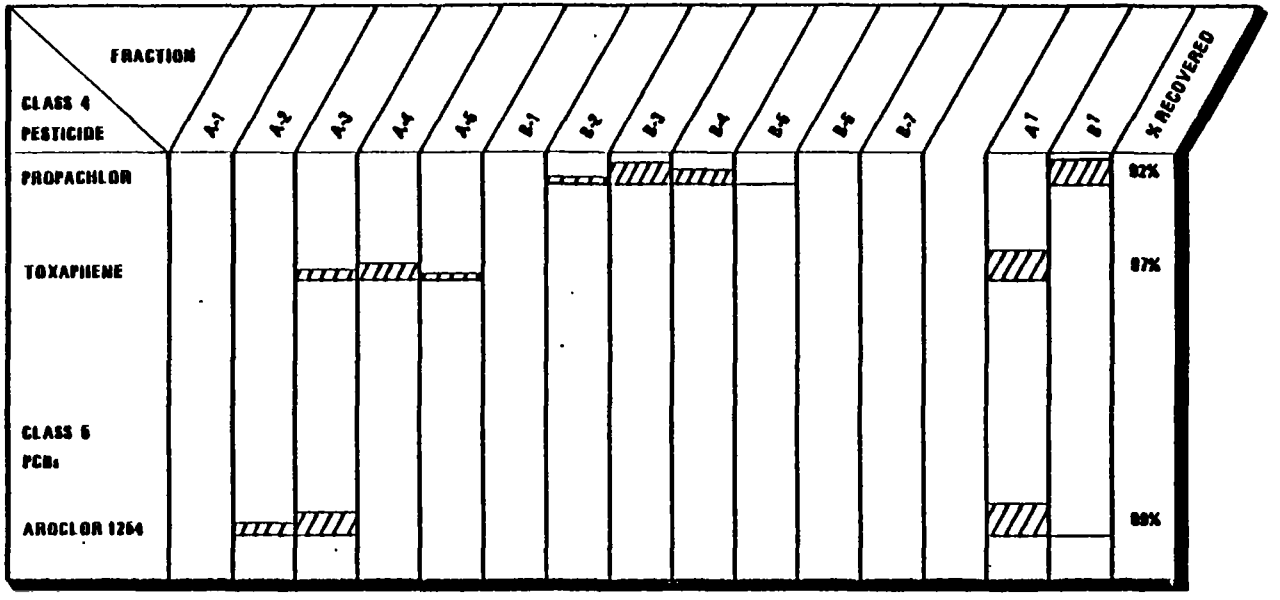


FIGURE 2-1
FLORISIL COLUMN PROFILE



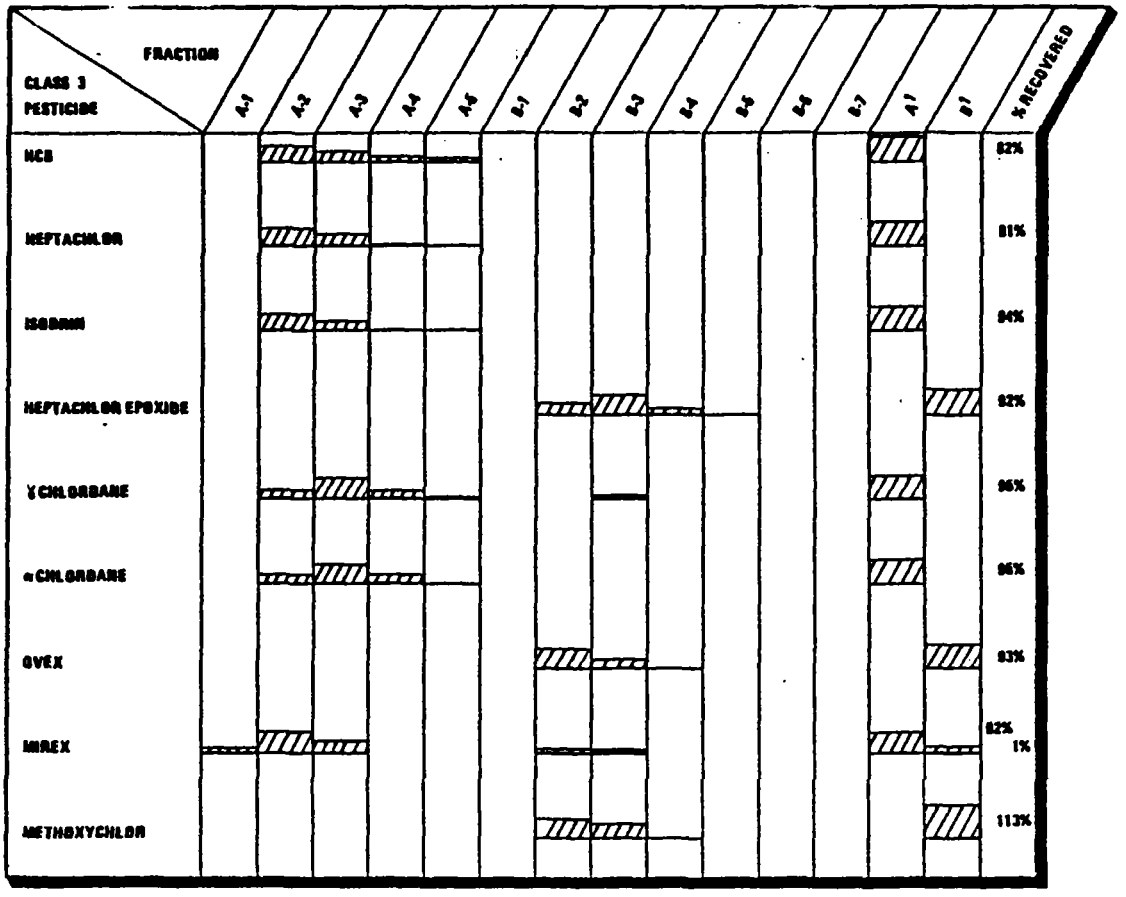
A = 100 ml OF 10% CH_2Cl_2 / 90% HEXANE (20 ml FRACTIONS)
B = 200 ml OF 100% CH_2Cl_2 (20 ml FRACTIONS)
A' = 100 ml OF 10% CH_2Cl_2 / 90% HEXANE COLLECTED AS ONE FRACTION
B' = 200 ml OF 100% CH_2Cl_2 COLLECTED AS ONE FRACTION

FIGURE 2-2
FLORISIL COLUMN PROFILE



A = 100 ml OF 10% CH_2Cl_2 / 90% HEXANE (20 ml FRACTIONS)
B = 200 ml OF 100% CH_2Cl_2 (20 ml FRACTIONS)
A' = 100 ml OF 10% CH_2Cl_2 / 90% HEXANE COLLECTED AS ONE FRACTION
B' = 200 ml OF 100% CH_2Cl_2 COLLECTED AS ONE FRACTION

FIGURE 2-4
FLORISIL COLUMN PROFILE



A = 100 ml OF 10% CH_2Cl_2 / 90% HEXANE (20 ml FRACTIONS)
B = 200 ml OF 100% CH_2Cl_2 (20 ml FRACTIONS)
A' = 100 ml OF 10% CH_2Cl_2 / 90% HEXANE COLLECTED AS ONE FRACTION
B' = 200 ml OF 100% CH_2Cl_2 COLLECTED AS ONE FRACTION

GLASSWARE WASHING PROCEDURE FOR TRACE ORGANICS GLASSWARE

The cleaning procedure for general glassware is as follows:

- Start with a hot soap and water wash contacting all glassware surfaces with a brush..
- Thoroughly rinse with hot tap water. Do not allow glassware to dry with soap residue.
- Soak glassware in hot RBS-35 surfactant solution for 30 minutes.
- Thoroughly rinse with hot tap water. Do not allow glassware to dry with surfactant still on it.
- Rinse interior of each piece of glassware with two portions of acetone (10-50 ml).
- Air dry and wrap openings with aluminum foil for storage.

The cleaning procedure for pipets is as follows:

- Thoroughly rinse pipet with appropriate solvent immediately after use.
- Place on RBS surfactant solution for soaking.
- Rinse with tap water via the automatic syphoning unit.
- Soak in hot RBS surfactant solution for 30 minutes.
- Rinse with tap water via the automatic syphoning unit.
- Rinse with acetone and air dry.

The cleaning procedure for miscellaneous pieces, i.e. teflon stockcocks, is as follows:

- Soak in RBS solution during the period of time between use and washing.
- Start with hot soap and water wash contacting all surfaces with a brush.
- Place on the dip rack of siphoning unit and rinse thoroughly with tap water.
- Soak in hot RBS solution for 30 minutes.
- Rinse again with tap water.
- Transfer pieces to large clean beaker and rinse 3 times with acetone and air dry.
- Rubber O-rings should not be acetone rinsed.

ATTACHMENT 6

PROCEDURES FOR PCBS IN SOILS/SEDIMENTS

4.0 EXTRACTION PROCEDURE FOR SOLID MATRICES

4.1 Thoroughly homogenize the sample by mixing with a stainless steel spatula. In some cases, it may be necessary to transfer the sample to a blender or ball mill to ensure complete homogenization.

4.2 Weigh a 30 gram portion into a tared pint extraction jar and add 60 grams of anhydrous sodium sulfate. Mix thoroughly with the spatula used to originally transfer the sample. The sample should have a sandy texture at this point. Immediately add 100ml of 1:1 methylene chloride/acetone to the sample.

Note: See Table 10, pg. 19 for detection levels and QA/QC requirements.

4.3 At this point, add the appropriate surrogate. For low level determinations add 100ul of the 20ppm dibutyl chlorendate stock prepared in methanol (2ug). For high level determinations, add 100ul of 300ppm DBC stock (30ug). Surrogates are added to blanks, spikes and samples.

4.4 Place the Teflon-lined cap on the extraction jar and shake vigorously. Place extraction jar in autoshaker and shake for thirty minutes. Allow the solid matrix to settle to the bottom, then decant the solvent extract into a second collection jar. You may prefer to transfer the extract using a disposable 25ml pipet. Repeat the extraction two more times with two additional 100ml portions of 1:1 methylene chloride and acetone. If a clear solvent layer is not achieved for each extraction, the entire extraction jar may be centrifuged between each extraction.

4.5 For low level analysis, pour the solvent extract from the collection jar through a funnel packed with sodium sulfate into a Kuderna-Danish (K-D) concentrator. Add 50ml of hexane to the collection jar and also pass it through the sodium sulfate into the K-D flask. Add 2 or 3 boiling chips, insert a 3-ball Snyder column, prewetted with hexane, and concentrate the extract to ±5ml on a steam bath. Proper evaporation is achieved when the balls in the Snyder column actively chatter, but do not flood with solvent. Remove the 3-ball Snyder column and add an additional 50ml of hexane and concentrate to ±2ml.

4.6 Disconnect the 10ml ampule from the K-D flask rinsing with small portions of hexane. Bring the final volume to 10ml with hexane. Transfer 1ml of extract to an auto-injection vial with ±.1g of activated copper metal. Transfer the remaining 9ml to a Teflon screw cap vial for storage.

4.7 For high level analysis, measure the extract volume in the collection jar, then transfer a 10ml portion to a 10ml ampule. Concentrate the extract to ±0.5ml on the nitrogen blowdown apparatus. Adjust final volume to 5ml with hexane. Transfer 1ml of the extract to an auto-inject vial with ±.1g of activated copper metal.

4.8 During the course of the sample extraction, add a quality control blank, matrix spike (MS) and matrix spike duplicate (MSD) to the batch or to every 20 samples. A blank is the glassware, reagents and surrogate (everything but the sample) and it is designed to monitor contamination from reagents or from the analysts' technique. To prepare a matrix spike and a matrix spike duplicate, select a sample randomly and weigh out 2 additional 30g portions into 2 extraction jars. At the point where surrogates are added spike the MS and MSD as follows:

Low Level Analyses		
Lindane	2ppm	
Heptachlor	"	
Aldrin	"	400ul
Dieldrin	5ppm	
Endrin	"	
4,4-DDT	"	

High Level Analyses	
PCB (any Aroclor)	300ug

4.9 Often soil matrices require some kind of cleanup before GC analysis. Four options are available: macro-florisil cleanup, micro-florisil cleanup, alumina column cleanup and acid wash.

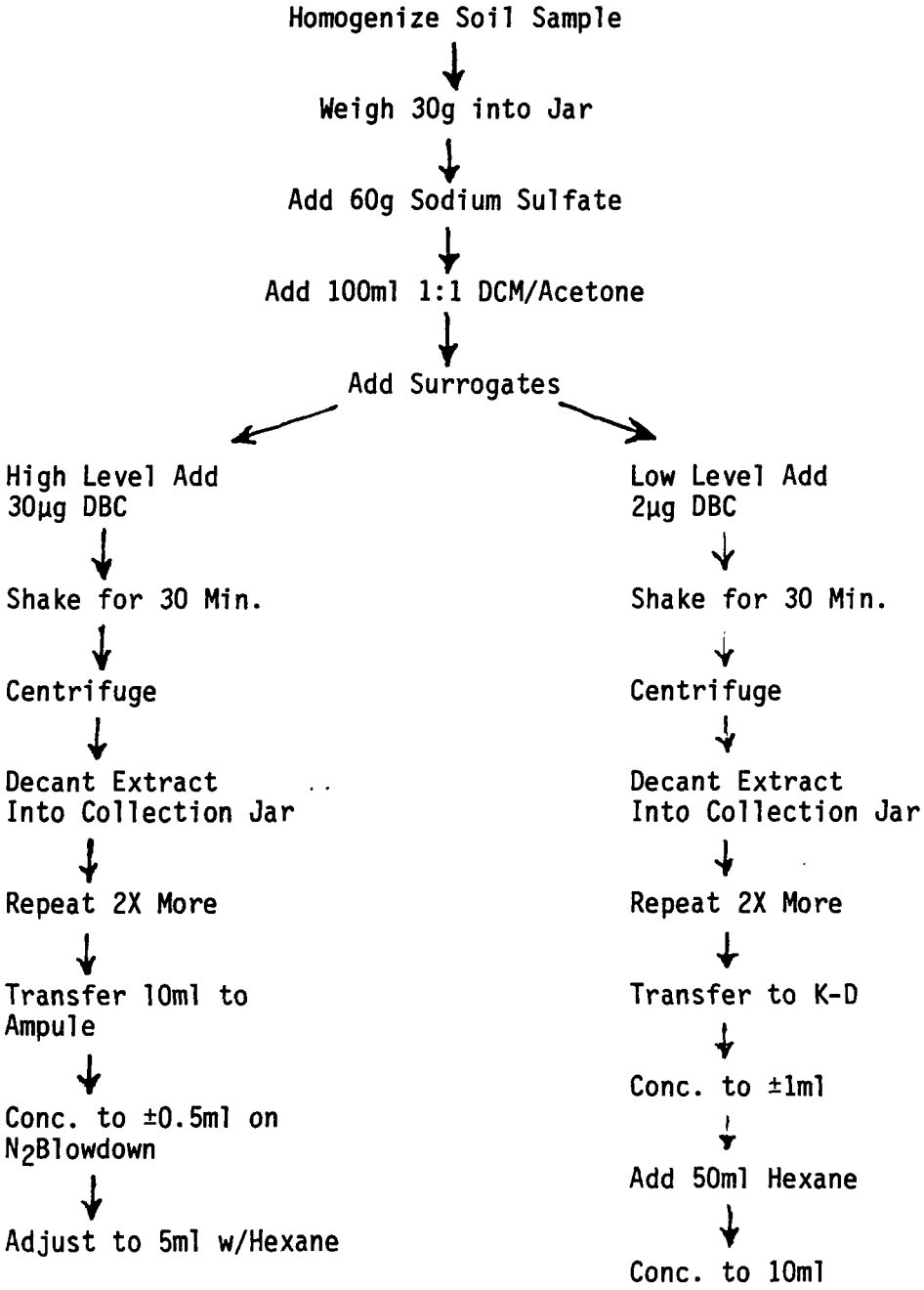
4.10 The detection limits for the high level and low level analyses are presented in the attached report.

PESTICIDE + PCBs

EXTRACTION PROCEDURE

FOR SOLID MATRICES

August 1986



4.0 EXTRACTION PROCEDURE FOR SEMI LOW SEDIMENTS

4.1 Thoroughly homogenize the sample by mixing with a stainless steel spatula. In some cases, it may be necessary to transfer the sample to a blender or ball mill to ensure complete homogenization.

4.2 Weigh a 30 gram portion (of dried sediment) into a tared pint extraction jar. Immediately add 100ml of 1:1 methylene chloride/acetone to the sample.

4.3 At this point, add the appropriate surrogate. For low level determinations add 100ul of the 20ppm dibutyl chlorendate or equivalent stock prepared in methanol (2ug). Surrogates are added to blanks, spikes and samples.

4.4 Place the Teflon-lined cap on the extraction jar and shake vigorously. Place extraction jar in auto-shaker and shake for thirty minutes. Allow the solid matrix to settle to the bottom, then decant the solvent extract into a second collection jar. You may prefer to transfer the extract using a disposable 25ml pipet. Repeat the extraction two more times with two additional 100ml portions of 1:1 methylene chloride and acetone. If a clear solvent layer is not achieved for each extraction, the entire extraction jar may be centrifuged between each extraction.

4.5 For low level analysis, pour the solvent extract from the collection jar through a funnel packed with sodium sulfate into a Kuderna-Danish (K-D) concentrator. Add 2 or 3 boiling chips, insert a 3-ball Snyder column, prewetted with hexane, and concentrate the extract to ±5ml on a steam bath. Proper evaporation is achieved when the balls in the Snyder column actively chatter, but do not flood with solvent. Remove the 3-ball Snyder column and add an additional 50ml of hexane and concentrate to ±2ml.

4.6 Disconnect the 10ml ampule from the K-D flask rinsing with small portions of hexane. Bring the final volume to 5ml with hexane. Transfer 1ml of extract to an auto-injection vial with ±.1g of activated copper metal. Transfer the remaining 9ml to a Teflon screw cap vial for storage.

4.7 During the course of the sample extraction, add a quality control blank, matrix spike (MS) and matrix spike duplicate (MSD) to the batch or to every 20 samples. A blank is the glassware, reagents and surrogate (everything but the sample) and it is designed to monitor contamination from reagents or from the analysts' technique. To prepare a matrix spike and a matrix spike duplicate, select a sample randomly and weigh out 2 additional 30g portions. At the point where surrogates are added spike the MS and MSD as follows:

	High Level Analyses
PCB (any Aroclor)	15ug

4.8 Often soil matrices require some kind of cleanup before GC analysis. Four options are available: macro-florisil cleanup, micro-florisil cleanup, alumina column cleanup and acid wash.

4.9 The detection limits for the high level and low level analyses are presented in the attached report.

DETECTION LIMITS

	WATER	SOIL(L)	SOIL(H)
PCB-1221	5	40	1000
PCB-1232	5	40	1000
PCB-1016/1242	5	40	1000
PCB-1248	5	40	1000
PCB-1254	5	80	2000
PCB-1260	5	80	2000

Units of Quantitation

Water = ng/l
Soil = µg/kg wet weight

ATTACHMENT 7

PROCEDURES FOR LOW-LEVEL PCBS IN WATER

5.0 EXTRACTION PROCEDURE FOR LOW LEVEL PCBs IN WATER

5.1 Determine pH of sample and adjust to a range of 5-9 with .1:1 sulfuric acid solution or in sodium hydroxide. Transfer the entire contents of sample bottle into a 2 liter separatory funnel.

5.2 Add 1ml of 50ppt DBC or equivalent (compound which will not be removed by cleanup options) surrogate solution to the sample.

5.3 To every batch of samples for low level analysis, add a blank, a matrix spike (MS) and a matrix spike duplicate (MSD). The blank is 2000ml of organic-free water and it is treated in the same manner as the samples. The MS and MSD samples are spiked in duplicate. Ideally, a sample is collected in triplicate in the field in 3 separate containers. The first sample is the sample itself. The second and third samples are spiked with the following compound in acetone:

Note: See Table 10, pg. 6 for detection levels and QA/QC requirements.

Aroclor 1254	5ppt

5.4 Add 50ml of 15% methylene chloride in hexane to the sample jar, seal and shake. Transfer bottle extract to separatory funnel and extract the sample by shaking vigorously for 2 minutes with periodic venting to release pressure.

5.5 Drain water sample back into sample jar. Drain the hexane extract through a sodium sulfate funnel into a Kuderna-Danish evaporator. Return water sample back to separatory funnel and repeat extraction 2 more times combining all extracts into the Kuderna-Danish evaporator.

5.6 If emulsion problems occur during extraction, collect all 3 extracts in a 200ml centrifuge bottle without sodium sulfate drying. Centrifuge the contents of the bottle until 2 distinct layers are formed. Transfer the top layer (the hexane extract) through sodium sulfate into the Kuderna-Danish evaporator.

5.7 Concentrate extract to 0.2ml.

5.8 If cleanup is necessary, refer to Section 7.0

5.9 Concentrate final extract after cleanup to 0.2ml and analyze by GC/ECD.

LOW LEVEL PCBs

EXTRACTION PROCEDURE

FOR WATER MATRICES

August 1986